QUALITY ASSURANCE PLAN / MANAGEMENT PLAN

PRAIRIE ANALYTICAL SYSTEMS, INC.

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James R. Johnson
President / Quality Assurance Officer

Stephen R. Johnson
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Revision No. 7
July 1998

1.0 General

1.0 General (cont.)

1.0 General (cont.)

2.0 General Policy Statement

2.1 Introduction

Prairie Analytical Systems, Inc. is an analytical testing laboratory established to provide a wide range of clientele with high quality analytical laboratory services. With the capability to provide both qualitative and quantitative analytical on a wide variety of matrices, including but not limited to liquid, solid and air, samples are received from both public and private sectors and groups organized to provide protection and preservation of the environment. These samples are analyzed in accordance with required and approved methodologies established by USEPA, AWWA, ASTM and other regulatory agencies.

The mission of the company is to provide services with demonstrable quality in a manner that meets all regulatory mandates. In order to satisfy and accomplish this mission, the company has developed a policy that includes a detailed quality assurance plan that has been implemented as an integral part of the operations and management of the laboratory.

As part of the high quality service offered by Prairie Analytical Systems, Inc., the laboratory places significant emphasis on the timeliness and accuracy of results. Much of the work is front-line monitoring or identification affecting the decision process by consultants, government and other private and public entities. Nearly all decisions impacting the fates of certain wastes, environmental and health threats or hazards associated with materials are based on the data produced by the analytical laboratory.

Our staff of professionals is committed to high quality service. The quality assurance program adopted by the company describes the practice of the laboratory as well as the implementation, management and review of these practices.

Many analytical procedures are under the USEPA guidance, e.g. contract lab program, and have well defined quality control practices associated with them. However, there are many others that do not fall under the umbrella of federal regulation and do not include well-defined quality control practices but are of no less importance to the clientele. It is, therefore, our goal to have acceptable and measurable checks of quality for all laboratory activities. When standard practices are not established, we make it our responsibility to develop and implement quality control practices, including activities affecting field sampling and measurement, which are not normally routinely considered in a Quality Assurance Plan.

The laboratory has received certification by the Illinois Environmental Protection Agency for the analysis of environmental samples under 35 IAC Part 186: Environmental Protection. The laboratory is certified for volatile, semi-volatile, element, and other wet chemistry methodologies as described in 35 IAC 186. The laboratory has also been certified for testing by the IA Department of Natural Resources.

2.2 The Quality Assurance Plan/Quality Control Document

As a systematic approach to quality, good laboratory practices are developed and implemented that document general techniques used in sample and equipment manipulation.

This document describes the Prairie Analytical Systems, Inc. Quality Assurance Plan fundamental to our activities and utilized in providing services as an analytical laboratory. It is important to have a unified approach to producing high quality data, regardless of clientele requirements. Therefore, regulatory requirements, sample type and parameter, and certain quality control practices are followed at all times without deviation.

All aspects of the quality assurance plan are monitored and audited by the quality assurance officer. This individual is also responsible for verifying that quality assurance goals are being met and advising laboratory personnel of the results of the monitoring and auditing.

2.3 Review of Quality Assurance Plan[1]

The laboratory management reviews t the QAP to ensure the QAP's continuing suitability, effectiveness and compliance with any accreditation requirements. The laboratory incorporates all changes, including, but no limited to: changes in approved test methods, changes in laboratory equipment, or changes in laboratory personnel. Upon any revision done to the formal Quality Assurance Plan, a form **PAS-QMR186.185 (f)** (see Page 2-3) must be completed and signed by the Laboratory Director and the Quality Assurance Officer. This form will document any deficiencies and corrective actions taken to the QAP by Section and Subsection. Once the QAP has been updated, approved and signed, a PAS-QMR 186.185 (f) is incorporated into all future QAP's until another revision is necessary.

QAP Management Review

Prairie Analytical Systems, Incorporated

QAP Revision No. 7 Date 01 March 99

4.3 (1) - James R. Johnson, QA Officer

Signature [signature] Initials [initials]

4.3 (2) - Stephen R. Johnson, Laboratory Director

Signature [signature] Initials [initials]

Section 1.0 - General	Accepted [✓]	Rejected [ ]
Section 2.0 - General Policy Statement	Accepted [✓]	Rejected [ ]
Section 3.0 - Laboratory Facility, Equipment and Materials	Accepted [✓]	Rejected [ ]
Section 4.0 - Chart of Organization and Responsibility	Accepted [✓]	Rejected [ ]
Section 5.0 - Sample Collection, Preservation and Storage	Accepted [✓]	Rejected [ ]
Section 6.0 - Sample Acceptance, Receipt and Tracking	Accepted [✓]	Rejected [ ]
Section 7.0 - Quality Assurance Objectives	Accepted [✓]	Rejected [ ]
Section 8.0 - Internal Quality Control Procedures	Accepted [✓]	Rejected [ ]
Section 9.0 - Analytical Methodology	Accepted [✓]	Rejected [ ]
Section 10.0 - Data Reduction, Validation & Reporting	Accepted [✓]	Rejected [ ]
Section 11.0 - Internal Laboratory Audits	Accepted [✓]	Rejected [ ]
Section 12.0 - Corrective Action	Accepted [✓]	Rejected [ ]
Section 13.0 - Management Records	Accepted [✓]	Rejected [ ]
Section 14.0 - Customer Relations	Accepted [✓]	Rejected [ ]
Section 15.0 - Bibliography	Accepted [✓]	Rejected [ ]

Section _____ Subsection _____

Deficiency:
Corrective Action:

Section _____ Subsection _____

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Corrective Action:

Comments:

3.0 Laboratory Facility, Equipment and Materials

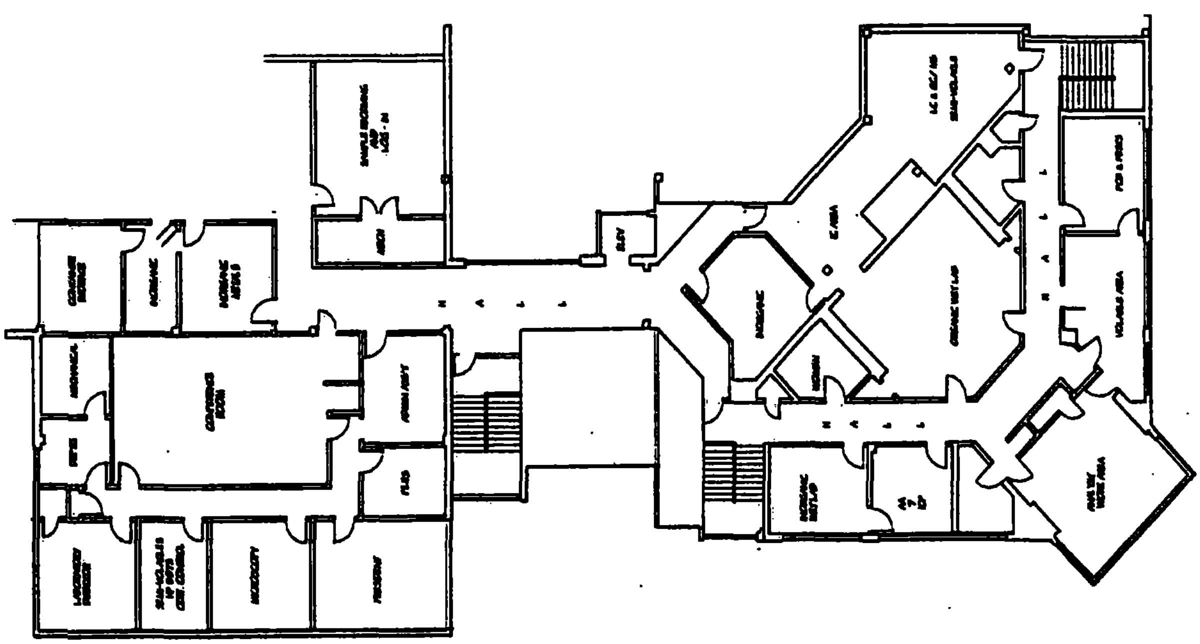
3.1 The Facility

The general offices and laboratory of Prairie Analytical Systems, Inc. are located at 1265 Capital Airport Drive, Springfield, Illinois 62707-8490, occupying 6743 square feet of administration and laboratory space. The area is divided into work zones, physically separated by walls, hallways, etc. to accommodate the sample organic and inorganic preparation areas, the inorganic analysis area, the volatile analysis area, the semi-volatile analysis area, the sample storage area and the administrative/office area. See Figure 3.1 below. The facility is arranged to provide complete separation of the organic and inorganic extractions and glassware washing areas from the GC/GC-MS and other instrumentation areas to help insure contamination free work areas. A total of 145 lineal feet of bench space is available in the sample preparation areas, and a total of 140 lineal feet of bench space is provided in the analytical areas.

Samples are received in the general administrative area and are identified on a properly completed chain of custody form, either checked in with the sample or completed by owner and laboratory personnel at the time of delivery. Each sample is then assigned a unique laboratory number. Once received and accepted by the laboratory, a project or work file is prepared with the appropriate worksheets and internal control data sheets, the sample is then placed in an approved cooler for sample storage until required for extraction and/or testing.

Waste storage is provided in properly identified vessels located in a remote ancillary structure on airport property approximately 1000 feet from the laboratory facility. The location of the building has been given a waste generator number by the Illinois Environmental Protection Agency. All wastes are disposed under the provisions of a contract between ChemWaste, Inc. and PAS, Inc.

Figure 3.1 - Laboratory Floor Plan



3.2 Laboratory Equipment

The primary analytical instrumentation consists of the following instruments or equipment listed by the area within the facility:

1. Volatile Organic Area

Hewlett-Packard 5890 Series II Gas Chromatograph with Tandem OI Analytical 5240 Photoionization Detector/Electrolytic Conductivity Detector (PID/ELCD); Capillary Injectors; TekMar 3000 Purge and Trap with Hand Held Controller; TekMar 2016-16 position Autosampler; Hewlett-Packard 3396B Integrator - Dual Channel.

Hewlett-Packard 5972 MSD Volatile System w HPIB includes: 5972 Detector; G1034C MS Software; IBM Compatible 486/66mhz Computer; 8MB RAM; 430 MB Hard Drive; 3.5" Floppy Drive; 120MB Tape Back-up; Sony VGA Color Monitor; keyboard and mouse; HPIB; Laser Jet 4 Printer; HP Ion Gauge Controller; HP Enviroquant Software; HP NIST Library; HP 5890 Series II Gas Chromatograph; Packed Injector; Jet Separator Makeup Gas Kit; Tekmar 3000 Purge and Trap Purge and Trap Concentrator; and a Tekmar Precept II Autosampler with heated purge pockets.

2. Semi-Volatile Organic Area

Hewlett-Packard 5890 Series II Gas Chromatograph with Electron Capture Detector (ECD) and Nitrogen Phosphorus Detector (NPD); split/splitless injectors; INET Communication; Pressure Regulators; HP 3396B integrator - Dual Channel.

Hewlett-Packard 5972 MSD/5890 Series II Gas Chromatograph Semi-Volatile System w/HPIB includes: 5972 Detector; G1034C MS Software; IBM Compatible 486/66mhz Computer; 8MB RAM; 430 MB Hard Drive; 3.5" Floppy Drive; 120MB Tape Back-up; Sony VGA Color Monitor; keyboard and mouse; HPIB; Laser Jet 4 Printer; HP Ion Gauge Controller; HP Enviroquant Software; HP NIST Library; HP 5890 Series II Gas Chromatograph; Split/splitless Injector with EPC, EPC Board, HPIB Communication; HP 7673 Autosampler - Single Tower.

Hewlett-Packard 5973 MSD/6890 Semi-Volatile System w/ECD includes: 5973 Mass Selector Detector, an Electron Capture Detector (ECD) w/EPC; G1036A NIST Chemical Library; 1038A Pesticide MS Spectral Library; G1020D MS ChemStation; HP Vectra XM5/150mhz computer; 32 MB Ram; 2.3 GB Hard Drive; 3.5" Floppy and CD ROM Drive; Mouse & Keyboard; HP Ultra VGA 1280 Monitor; H-P Laserjet 5 Printer; ion gauge controller; HP 6890 Series Injector/Autosampler; Split/Splitless Injector with EPC.

Hewlett-Packard 1050 HPLC System includes: HPLC Chemstation Software on H-P 586/100mhz, 16MB RAM, 540 MB Hard Drive Computer, 17" VGA Monitor, keyboard & Mouse; w/Quaternary pump, programmable sampler, diode-array detector, programmable fluorescence detector, and PAH Column 5um & Hypersil ODS-5 Column.

3. Wet Chemistry & Inorganic Area

Hewlett Packard 4500 ICP/MS, w/HP Vectra Pentium PC and ICP/MS Software; sample probe wash pump; CETAC auto-autosampler; air-cooled non-CPC water chiller. NESLAB CFT-75 Refrigerated Recirculator.

Perkin-Elmer 4100ZL Atomic Absorption Spectrometer with Fume Extractor and Cooling System and 4100ZL System Controller Assembly, 2 lamp EDL Power supply. IBM.

Perkin-Elmer Plasma 400 ICP, P-400, Controller, P-400 Software. Okidata 320 Printer, Perkin-Elmer AS-90 Autosampler and Controller. Digital Celebus 466 Computer.

Perkin-Elmer 1600 Series FTIR w/HP Colorpro Plotter

Dionex DC-120 Ion Chromatography w/Dual Column and 4400 Integrator, AS40 Automated Sampler.

Coleman Mercury Analyzer, Model 50B

Orion Model 920A pH/ISE Meter with 900A Printer

Orion Model 290A pH/ISE meter

Orion Model 124 Conductivity/TDS Meter

Sartorius Model BP211D 5-place balance with computer/printer read-out

Sartorius Model BA61 Toploading Balance

Sartorius Model B1417-93 Analytical Balance

Sartorius Model LC420 Analytical Balance

Sequoia-Turner Model 340 Digital Spectrophotometer

Sonics and Materials Model VC375 Ultrasonic Processor

3M Manifold w/147MM SPE Reservoir

Barnstead Nanopure Infinity UV/UF Water Purification System

Barnstead Model D0800 Water Purification System

Barnstead Model Epure Ultrapure Water System (4-module)

Gelman Pressure Filter

Environmental Express Tumbler Model GFM060JI

Rosemount/Dohrman Automated DC-190 Total Organic Carbon Analyzer

Tekmar-Dohrman DH-DX-2002 Organic Halide Analyzer (TOX, EOX) w/AD 2000 Adsorption Module.

Zeiss Axioskop (Phase Contrast, Polarized Light, Brightfield and Darkfield capability)

Zeiss Stemi-200C Stereoscope w/Schott KL-1500 Fiber Optic Illuminator.

4 - Revco Model R134A Cryo-Fridge Sample Coolers

6 - Electrothermal Uni-Mantles

VWR Model 2005 Low-Temp Incubator

Thermolyne Model 1400 Furnace

Baxter Model DX-31 Drying Oven

Precision Scientific Conc-Ring 12 Water Bath

Precision Scientific Pensky-Martin Closed Cup Flashpoint Apparatus

Labconco six foot Safeaire fume hood

Labconco four foot Safeaire fume hood

3 - Holiday Standard freezers

ELE K-605A Combination permeameter

(All instruments and equipment purchased as new equipment)

Major instrumentation is maintained under service agreements with the manufacturers. Maintenance is also performed by the laboratory director and documented in a designated Equipment Maintenance Log Book.

3.3 Equipment Maintenance[1]

The responsibility of the routine care and maintenance of equipment and instruments lies with the laboratory director. Maintenance is performed on instruments on an as needed basis when the quality control of a method cannot be met. Installation and maintenance activities are kept on file for reference in the Equipment Maintenance Logbook. Repairs that cannot be performed by the in-house staff are performed by manufacturers' service personnel. Analytical balances are checked annually under the provisions of a service contract. Equipment maintenance that is performed on a regular schedule is done as follows:

1. Analytical Balance - balances with a sensitivity of at least 0.1 mg.

    a. The laboratory checks each analytical and pan balance monthly with a minimum of two ASTM type 2 weights covering the effective range of the balance's use.

    b. The laboratory has a current service contract in effect on all analytical balances.

        1. The balances are serviced annually by a qualified service representative.

        2. The laboratory retains a certificate supplied by an authorized service representative which identifies traceability of the calibration to NIST standards.

2. pH Meter - pH meters having the accuracy of at least +/- 0.1 pH Units and a scale readability of at least 0.1 pH Units.

    a. The laboratory utilizes an automatic compensation device to correct pH measurements according to the current temperature.

    b. The laboratory calibrates the pH meter before each use with a minimum of two standardization buffers in the appropriate pH range.

    c. If linearity is out of control, the laboratory replaces the electrode.

3. Conductivity meter - a conductivity meter with an error not exceeding 1% or one µmhos/cm whichever is greater.

a. The laboratory calibrates the conductivity meter before each use.

b. The laboratory calibrates the conductivity meter with a standard that reflects conductivity.

4. NIST Thermometer - NIST Traceable thermometer with 1 °C or finer subdivisions and a range which spans the various requirements of the analytical method

a. The laboratory ensures that the thermometer is calibrated at least once every five years.

b. The laboratory retains a certificate identifying the traceability of the calibration to the NIST standard.

5. Thermometer - thermometer with 1 °C or finer subdivisions and a range which spans the various requirements of the analytical method

a. The laboratory calibrates all thermometers against an NIST traceable thermometer once annually and use the calibration factor for continued use.

6. Refrigerator - each refrigerator shall be identified in a way which establishes its use and distinguishes it from the others.

a. The laboratory monitors daily the temperature of each refrigerator. Sample refrigerator that store samples that require thermal preservation at 4 °C shall be between 0.1 - 6.0 °C. All other shall be +/- 2 °C of the specified temperature.

b. If temperature is erratic, the laboratory calls a service representative.

7. Freezer - each freezer shall be identified in a way which establishes its use and distinguishes it from the others.

a. The laboratory monitors daily the temperature of each freezer. Freezer temperature shall be -15 +/- 5 °C.

b. If temperature is erratic, the laboratory calls a service representative.

8. Oven - each oven shall be identified in a way which establishes its use and distinguishes it from the others.

a. The laboratory monitors the temperature each day of use and insures compliance with the specific method requirements.

b. Until the temperature is stabilized to meet the method specifications, no analysis will be run.

c. If temperature is erratic, the laboratory calls a service representative.

9. Incubator - each incubator shall be identified in a way which establishes its use and distinguishes it from the others.

a. The laboratory monitors the temperature each day of use and insures compliance with the specific method requirements.

b. Until the temperature is stabilized to meet the method specifications, no analysis will be run.

c. If temperature is erratic, the laboratory calls a service representative.

10. Pure Water Source - the laboratory has available a source of distilled and deionized water.

a. The laboratory records daily the conductivity and insures resistivity values of at least 0.5 megohm-cm at 25 °C.

b. Because the laboratory is utilizing an in-line conductivity meter for the daily check, the meter is calibrated monthly.

c. When the laboratory is using an external source for measuring conductivity, the laboratory shall collect the water from a frequently used access point.

d. If the resistivity value does not met the above requirement, the problem is identified and corrected.

11. Graphite Furnace

a. The windows are washed with alcohol.

b. The optical sensor is washed with alcohol and checked for pitting or excessive wear.

c. The contact cylinders are replaced as required.

12. ICP and ICPMS

a. The torch is cleaned in aqua regia as needed.

b. The spray chamber washed with soapy water as needed.

c. Nebulizer is cleaned as needed.

d. Waste system is empty as needed.

e. System is cleaned and optimized by service representative annually.

13. Gas Chromatograph

a. The septum is replaced weekly.

b. The inlet liner is replaced as needed.

c. The detector is cleaned and checked as needed.

14. Gas Chromatograph - Mass Selective Detector

a. The septum is replaced weekly.

b. The inlet liner is replaced as needed.

c. The detector is cleaned and checked as needed.

d. The vacuum pumps oil is changed semi-annually.

All quality assurance plan checks referenced above are recorded daily by a laboratory technician on form **PAS-QAPC 186.145** (see Page 3-8).

3.4 Laboratory Materials[1]

All glassware used for the purpose that may subject it to damage from heat or chemicals are made of borosilicate glass. All volumetric glassware are ASTM class A.

The laboratory utilizes analytical standards that are traceable to a national standard where available. The laboratory utilizes analytical reagents of reagent grade (AR) or better. The laboratory documents and maintains records (see form **PAS-SLOGIN186.190 (f)** Page 3-9) concerning the receipt, use, and traceability of analytical standards and reagents and includes:

1. verification that standards are traceable to national standards. If traceability to a national standard is not possible, the laboratory demonstrates, by an appropriate means (e.g. analyses of PE samples) that the instrumentation and equipment is properly calibrated;

2. certificate of origin, purity and traceability of all standards and reagents. These records include the date of receipt, storage conditions, the date of opening and expiration date;

3. procedures to ensure the traceability of working and intermediate standards to purchased stock standards or neat compounds which include the date of preparation and preparer's initials; and

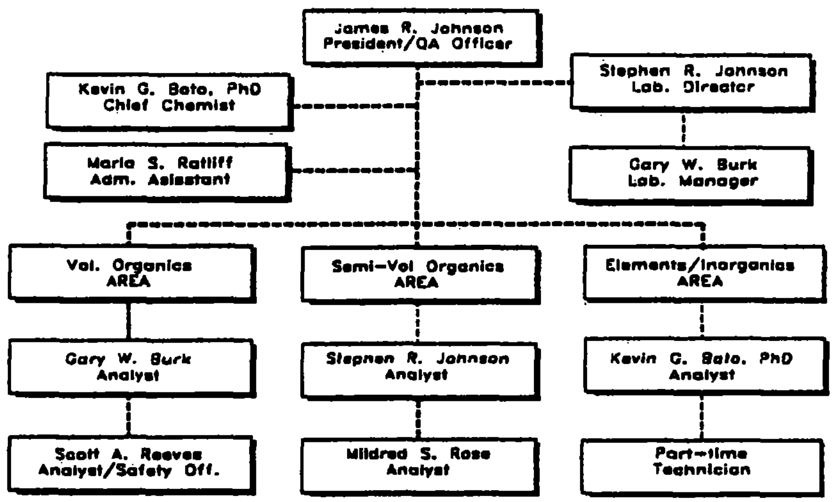
4. procedures to clearly identify all prepared reagents and standard, including: preparation date, concentrations and preparer's initials.

4.0 Chart of Organization and Responsibilities

4.1 Organization Chart

The responsibilities for insuring compliance with the QAP are distributed to the various professionals employed by the company. The Organization of the Company is as shown below.

Figure 4.1 Organization Chart



4.2 Personnel Requirements[1]

1. The laboratory owner has designated one individual as laboratory director. The laboratory director (see 4.3.2) :

   a. holds a minimum of a bachelor's degree in natural or physical sciences and has completed enough course work in chemistry to equal a minor in chemistry;

   b. has a minimum of two years experience managing a laboratory;

   c. is an employee of the laboratory; and

   d. is responsible for:

      1. analytical and operational activities of the laboratory;

      2. supervision of personnel employed by the laboratory;

      3. assuring that sample acceptance criteria are met, that samples are logged into the sample tracking- system that samples are properly labeled and that samples are properly stored;

      4. the production and quality of data reported by the laboratory;

      5. designating laboratory supervisors; and

      6. designating at least one individual as the quality assurance officer.

2. The laboratory owner or director has designated at least one individual as laboratory supervisor. The laboratory supervisor (see 4.3.4):

a. holds a minimum of a bachelor's degree in natural or physical sciences and has completed enough course work in chemistry to equal a major in chemistry;

b. has a minimum of one year of experience in the analyses pertaining to the applicable fields of testing;

c. is an employee of the laboratory; and

d. is responsible for:

1. supervising analysts, analysts-in-training and technicians in the area of analytical responsibility;

2. reviewing and verifying data produced by an analyst-in-training; and

3. reviewing and verifying(, data produced by a technician.

3. The laboratory owner has not designated a laboratory supervisor as laboratory director. The laboratory director/supervisor must fulfill the requirements of subsections (a)(2) and (4) and (b).

4. The laboratory director has designated one individual as the quality assurance officer. The quality assurance officer (see 4.3.1):

a. holds a bachelor's degree in natural or physical sciences and has completed enough course work in chemistry to equal a major in chemistry;

b. has a minimum of one year experience as an analyst in a laboratory and has documented training in quality assurance and quality control (QA/QC);

c. where applicable, has functions independent from laboratory operations;

d. has a general knowledge of the analytical methods for which data review is performed;

e. is an employee of the laboratory; and

f. is responsible for:

1. coordinating QA/QC procedures and analytical data review procedures in the laboratory;

2. verifying that the requirements in Section 186.160 of this Part are met; and

3. conducting internal audits of the entire laboratory operation annually.

5. The laboratory director has designated the analysts. Analysts (see4.3.2, 4.3.3, 4.3.4, 4.3.5, 4.3.6):

a. hold a bachelor's degree in natural or physical sciences and have completed enough course work in chemistry to equal a major in chemistry;

b. have a minimum of one year experience in the analyses pertaining to the applicable fields of testing for which the laboratory has and is seeking accreditation;

c. for those instruments listed in subsection (g) below:

1. either:

i. have satisfactorily completed a minimum of four hours training that is offered by the equipment manufacturer, a professional organization, a university or another qualified training facility; or

ii. served a two-week period of apprenticeship under an experienced analyst; and

2. have on file documentation indicating acceptable performance on a blind sample at least once per year and a certification that the analyst has read, understood and agreed to perform the most recent version of the *method, the approved method or standard operating procedure.* Such documentation shall demonstrate that the required training is up-to-date;

d. after appropriate training pursuant to subsection (5)(c), perform the IDMP study, as specified in 35 IAC 186.160;

e. are an employee of the laboratory, contract employee or contracted temporary agency staff; and

f. are responsible for reviewing and verifying data produced by analysts-in-training or technicians when a laboratory supervisor does not review and verify the data.

6. The laboratory directors or supervisors may designate individuals as analysts-in-training. Analysts-in-training must at least meet the requirements in subsection (8) and must be in the process of meeting the requirements of subsection (5). A laboratory supervisor or analyst shall review and verify all data produced by analysts-in-training.

7. Analyses performed utilizing Atomic Absorption (AA), Ion Chromatograph (IC), Gas Chromatograph (GC), Gas Chromatograph/Mass Spectrometer (GC-MS), Inductively Coupled Plasma (ICP), Inductively Coupled Plasma/Mass Spectrometer (ICP-MS), Direct Current Plasma Spectrometer (DCP), Liquid Chromatograph/Mass Spectrometer (LC-MS), High Pressure Liquid Chromatograph (HPLC), or Transmission Electron Microscope (TEM) are only acceptable for the purposes of this 35 lac 186 when performed by a laboratory employee who meets the requirements in subsection (5) or (6) above.

8. A technician is a person who holds a minimum of a high school diploma or its equivalent. A technician must:

a. either:

1. have satisfactorily completed a minimum of four hours training that is offered by the equipment manufacturer, a professional organization, a university or qualified training facility; or

2. served a two-week period of apprenticeship under an experienced analyst or technician;

b. after appropriate training, pursuant to subsection (8)(a), perform the IDMP study, as specified in 35 IAC 186.160; and

c. have on file documentation indicating acceptable performance on a blind sample at least once per year and a certification that the technician has read, understood and agreed to perform the most recent version of the method, the approved method or standard operating, procedure. Such documentation shall demonstrate that the required training is up-to-date.

9. If a person serves in any capacity as defined in subsections (a) through (h) and that person does not meet the training, educational or experience requirements for the position. The laboratory will submit written justification to the Agency explaining why a laboratory director, laboratory supervisor, quality assurance officer, analyst, analyst-in-training, or technician should serve in that position. The written justification shall take into account the following factors:

a. either:

1. experience as an offset for educational requirements (such as, one year of experience performing the applicable duties equals one year of education);

2. education as an offset for experience requirements (such as, one year of applicable education beyond a bachelor's degree equals one year of experience);

3. for the quality assurance officer, have six month's experience in quality assurance and quality control procedures and be knowledgeable in the quality systems as defined under this Part as an offset for the training requirements specified in subsection (4)(b); or

4. for analysts and technicians, have six months laboratory experience as offset for the training and apprenticeship requirements set forth in subsections (5)(c)(1) and (2), (8)(a) and (8)(b). Laboratory experience must be in the analytical technique for which the offset is requested.

b. for analysts and technicians, demonstration of ability to properly perform representative test procedures.

4.3 Personal Data for Key Personnel

1. James R. Johnson

a. Education - BFA in LA - University of Illinois 1961
Post graduates studies in socio-ecology and terrestrial eco-systems

b. Position - President and Quality Assurance Officer

c. Experience - Mr. Johnson has accumulated thirty-eight years of experience in the field of landscape architecture and environmental studies. For nine years, he was employed by the State of Illinois as the District Landscape Architect for District 6 of the Illinois Department of Transportation where a portion of his responsibility was evaluating the effects of new pesticides and herbicides on the flora and fauna associated with highway roadsides. In 1972, Mr. Johnson was named as a special advisor to the Governor of the State of Illinois and the Argonne National Laboratories on matters dealing with the reclamation of abandoned mined lands in Illinois.

From 1973 to 1993, Mr. Johnson was a partner in a consulting engineering firm that specialized in environmental assessments, water and sewer plant design and development, mine reclamation, drainage studies, and landscape architecture. During mixed, held for sixteen hours and verified to be pH<2 prior to withdrawing an aliquot for analysis. For the determination of dissolved elements, the sample must be filtered through a 0.45 µm pore diameter membrane filter at the time of collection or as soon thereafter as practically possible. Acidify with 50% HNO_3 immediately following filtration to pH <2. Solid samples require no preservation other than storage of 4°C

c. Holding time.

1. Samples must be preserved within 14 days and digests analyzed within six months.

d. Field blanks

1. Processing of a field reagent blank (FRB) is recommended along with each sample set, which is composed of the samples collected from the same general sample site at approximately the same time. At the laboratory, fill a sample container with reagent water, seal, and ship to the sample sate along with the empty sample containers. Return the FRB to the laboratory with the filled sample bottles.

5. Sample collection, preservation, and storage - **Method OA-1**

a. Sample collection, dechlorination, and preservation.

1. Collect all samples in duplicate. Samples shall be collected by a person that has been certified by the Iowa Department of Natural Resources (DNR as specified by IAC Chapter 135 (DNR). If samples contain residual chlorine, and measurements of the concentrations of disinfection by-products (trihalomethanes, etc.) at the time sample collections are desired, add about 25 mg of ascorbic acid (or 3 mg of sodium thiosulfate) to the sample bottle before filling. Fill samples to overflowing, but take care not to flush out the rapidly dissolving ascorbic acid (or sodium thiosulfate). No air bubbles should pass through the sample as the bottle is filled, or be trapped n the sample when the bottle is sealed. Adjust the pH of the duplicate samples to <2 by carefully adding one drop of 1:1 HCl for each 20ml of sample volume. Seal the sample bottles, PFTE-face down, and shake vigorously for 1 min.

2. When sampling from a water tap, open the tap and allow the system to flush until the water temperature has stabilized (usually about 10 min). Adjust the flow to about 500 ml/min and collect duplicate samples from the flowing stream.

3. When sampling from an open body of water, fill a 1-quart wide-mouth bottle or 1-liter beaker with sample from a representative area, and carefully fill duplicate sample from the 1-quart container.

4. The samples must be chilled to 4°C on the day of collection and maintained at that temperature until analysis. Field samples that will not be received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to ensure that they will be at 4°C on arrival at the laboratory.

b. Sample storage.

1. Store samples at 4°C until analysis. The sample storage area must be free of organic solvent vapors.

2. Analyze all samples within 14 days of collection. Samples not analyzed within this period must be discarded and replaced.

c. Field reagent blanks.

1. Duplicate field reagent blanks must be handled along with each sample set, which is composed of the samples collected from the same general sample site at approximately the time. At the laboratory, fill field blank sample bottles with reagent water, seal, and ship to the sampling site along with empty sample bottles and back to the laboratory with filled sample bottles. Wherever a set of samples is shipped and stored, it is accompanied by appropriate blanks.

2. Use the same procedures used for samples to add ascorbic acid (or sodium thiosulfate) and HCL to blanks (Section 5.5a, 1).

6.Sample Collection, Preservation, and Storage - **Method OA-2**

a. Sample collection.

1. Samples are collected by a person certified by the Iowa Department of Natural Resources (DNR) according to the provision specified in IAC Chapter 135 (DNR). Samples shall be collected in amber glass containers. Keep samples sealed from collection time until analysis. When sampling from an open body of water, fill the container with water from a representative area. Sampling equipment, including automatic samplers, must be free from plastic tubing, gaskets, and other parts that may leach analytes into the water. Automatic samplers that composite samples over time must use refrigerated glass sample containers.

b. Sample dechlorination and preservation.

1. All samples should be iced or refrigerated at 4°C from the time of collection until extraction. Residual chlorine should be reduced at the sampling site by addition of reducing agent. Add 40-50 mg of sodium sulfite or sodium arsenite (these may be added as solids with stirring until dissolved) to each liter of water. Hydrochloric acid should be used at the sampling site to retard the microbiological degradation of some analytes in unchlorinated water. The sample pH is adjusted to <2 with 6 N hydrochloric acid. This is the same pH used in the extraction, and is required to support the recovery of pentachlorophenol.

c. Holding time.

1. Samples must be extracted within 7 days and extracts analyzed within 14 days of sample collection.

d. Field blanks

1. Processing of a field reagent blank (FRB) is recommended along with each sample set, which is composed of the samples collected from the same general sample site at approximately the same time. At the laboratory, fill a sample container with reagent water, seal, and ship to

the sample sate along with the empty sample containers. Return the FRB to the laboratory with the filled sample bottles.

2. When hydrochloric acid is added to samples, use the same procedures to add the same amount to the FRB.

6.0 Sample Acceptance, Receipt and Tracking

6.1 Description of the chain-of-custody.

A sample is considered under custody if it is in the possession of Prairie Analytical Systems, Inc. and has met the acceptance criteria of the Sample Acceptance and Receipt Standard Operating Procedure. Sample custody is an integral and necessary part of any comprehensive quality assurance program. Since laboratory data is often used in evidence in the courts, it is imperative that the integrity of the sample is maintained from the time of collection to the time of data reporting. Standard custody procedures are outlined and identified below.

Chain-of-Custody initiates with the proper collection of samples at the site or point of origin. Custody for the lab begins when the sample and the proper chain-of-custody form has been received and accepted by a representative of Prairie Analytical Systems, Inc. A sample is considered to be in a person's custody if:

1. it is in that person's physical possession;

2. it is in the view of that person after he has taken possession of the sample; or

3. it is secured by that person in an area which is restricted to authorized personnel.

Persons who have samples under their custody must comply with the procedures described in the following section. Compliance shall be initiated at the point appropriate in the chain-of-custody scheme as identified, field custody, transfer of custody and in-laboratory custody, since custody initiates at the point of receipt and acceptance of the sample.

The record keeping shall commence immediately at the point where the sample is accepted by authorized personnel. Pertinent items of chain-of-custody shall require at a minimum:

1. complete documentation which shall include sample identification, the location, date and time of collection, sampler's name, preservative added, sample type and any special remarks concerning the sample;

2. sample labeling:

   a. a unique identification of the sample and each container; and

   b. a labeling system for the sample(s) with durable labels and use of indelible markings;

3. documentation of the use of preservative and sample containers as specified by the approved test methods;

4. adherence to the maximum allowable holding time prior to analyses as specified by the approved test methods; and

5. adequate sample volume to perform the necessary analyses.

6.2 Procedures

Only samples that provide a good representation of the media being sampled should be taken. The quantity of samples, the types of samples, and the sample locations are determined prior to any field work actually performed. All samples should be taken under the direct supervision of the field sampler for the project, with as few people as possible handling the samples.

The field sampler is personally responsible for the care and custody of the collected samples until they are transferred or dispatched properly.

Sample labels shall be completed for each sample using a waterproof ink, whenever possible. Labels shall be affixed to the sample containers prior to the actual collection of the sample and completed at the time of sampling.

The field supervisor determines whether proper custody procedures have been followed during the field work and makes the determination if additional samples are needed.

Sample seals shall be used on all samples if they are to leave the immediate and direct control of the field supervisor to detect unauthorized tampering. Sample seals may be a gummed paper seal or similar material. The paper seal, if required, shall contain the same information as appears on the sample label. The seal shall be attached in such a way that the seal must be broken in order to open the sample container.

6.3 Documentation

A chain-of-custody record shall be completed and accompany every sample to establish documentation to trace sample possession. A sample copy of this record is included with this QAP identified as form **PAS-COC 186.185 (b)** (See Page 6-7). This record shall contain the following minimum information:

1. client, address, phone and facsimile;
2. client project and project location;
3. sampler(s) name and telephone number;
4. sample(s) description;
5. sampling date and time;
6. container(s) size and quantity;
7. matrix and preservative code;
8. analysis and method requested;
9. PAS Sample Number;
10. sample acceptance or rejection;
11. signature of person(s) involved in chain of possession;
12. remarks regarding anything unique about sample(s);
13. method of shipment;
14. PAS Project Code.

6.4 Laboratory Operations[1]

The sample custodian is responsible for receiving all samples. Upon receipt of the sample, the custodian will verify the integrity of the sample to assure that the sample containers are not broken or compromised in any way, that the samples are properly identified and documented to the chain-of-custody and that the proper preservation of the sample has been provided and that the maximum holding time for each method has not been exceeded. The laboratory shall examine the samples for thermal preservation, if applicable. All samples which require thermal preservation shall be considered acceptable if:

1. the arrival temperature is either within +/- 2°C of the required temperature or the method specified range (for samples with a specified temperature of 4°C, samples with a temperature of 0.1 to 6 °C shall be acceptable); or

2. the sample has been hand delivered to the laboratory within six hours after collection and there is evidence, such as arrival on ice, that the chilling has begun.

Any samples that have been compromised or improperly preserved are to be identified by checking the rejected box on the PAS-COC and on the PAS-LOGIN. Data from any samples that does not meet the acceptance criteria must be flagged in an unambiguous manner clearly defining the nature and substance of the variation.

When the sample does not meet the preservation and maximum holding times requirements as stated in the approved test method, the laboratory shall notify the client requesting the analyses for further instructions before proceeding. If the sample does not meet the sample acceptance criteria the laboratory shall:

1. retain the correspondence and records of conversations concerning the final disposition of rejected samples; or

2. fully document any decision to proceed with the analysis of compromised samples including:

a. documenting the condition of the samples in the sample tracking records on the evidentiary chain of custody or transmittal form and the laboratory receipt documents; and

b. appropriately qualifying the analysis data on the final report.

Once sample integrity and protocol has been established, the samples will be recorded into the sample log-in book on form **PAS-LOGIN 186.185 (f)** (See Page 6-8). A laboratory identification number is assigned and attached to each sample container. Laboratory identification numbers are generated by the day of receipt plus a consecutive four-digit number, e.g. 9305310139 translates into the year 93, the month 05, the day 31, the sample number 0139.

Sample log-in sheets must include at a minimum a copy of the COC and the following items chronologically ordered:

1. date and time of laboratory receipt of sample;

2. sample collection date;

3. unique PAS Sample Number;

4. client sample description;

5. requested analysis (including approved test method number);

6. signature or initials of sample custodian;

7. comments resulting from the inspection for acceptance and rejection; and

8. PAS Project Code.

The sample custodian is responsible for sample security, accessibility and storage. Samples are stored according to preservation requirements (temperature, darkness, etc.), protected from cross-contamination, and are accessible for further analyses. Samples are stored as volatiles, semi-volatiles or inorganics. Locked storage is not required since the access is limited and controlled to the area where the samples are stored. All extracts and leachates will be properly stored and controlled. Standards are stored separately from the samples.

The sample custodian examines the samples upon receipt and a maximum holding time is given each work folder according to the parameters to be measured. The laboratory manager is responsible to review the work folders daily to insure that any holding time has not been exceeded. All extractable samples are to be extracted within 48 hours of the receipt of that sample to reduce storage problems and logistics scheduling.

The requested analytical test parameters are transferred from the chain-of-custody form and field book records to lab worksheets and an intralaboratory transfer sheet. Copies of a sample worksheet is included in this section (See Page 6-8). In each work folder there is an Intra laboratory Transfer Sheet form **PAS-ITS 186.190 (a) (3)** (See Page 6-9) that tracks the history of the sample, sample extracts and sample digests. The worksheets are checked to insure that the requested testing is identified and that the proper priority is assigned. The sample and the worksheet file is then delivered to the designated analytical area.

6.5 Sample Tracking[1]

Prior to analysis of a sample, the analyst performing the analysis must initial and date the ITS or receive the sample from the sample custodian, who will initial and date the ITS. Once the sample is in the hands of the chemist/analyst, he is responsible for tracking the sample through the testing process, and the remaining sample is placed in proper storage. The manager will then either approve the data as obtained or request additional data be gathered on the sample. Once the data is acceptable, the manager or director will sign the ITS and deliver the worksheets and the quality control data to the QAO. The QAO reviews and approves the quality control data before it is released to the data processor for preparation of the final report.

The approved data is forwarded to the data processor for the preparation of the data in a final format of a Certificate of Analysis report. Once completed, the report is checked and signed by the laboratory director and forwarded to the client as described in Section 10.0.

The laboratory documents and maintain records related to all procedures and activities to which a sample is subjected including:

1. identity of personnel involved in sampling, preparation and testing;

2. sample preservation, sample container and compliance to holding times;

3. sample identification code, receipt, log-in, acceptance and rejectance;

4. sample storage and tracking including: shipping receipts, transmittal forms and internal routing, internal laboratory transfer sheets and assignment records;

5. sample preparation including: cleanup and separation procedures, extract or digest identification codes, volumes, weights, instrument printouts, meter readings, calculations and reagents;

6. sample analysis;

7. equipment receipt, use, specification, operating conditions and preventative

maintenance;

8. calculations and statistical formulae used by the laboratory including

   a. written procedures for all calculations;

   b. representative calculations that indicate routine calculations;

   c. all raw data and supporting information needed to recreate calculation;

   d. appropriate number of significant digits are carried out throughout all recorded calculations; and

   e. the least precise step is identified in the calculation and the number of significant figures is an accurate reflection of the actual tolerances of the instrument or equipment;

9. procedures to verify that the reported data is free from transcription and calculation errors;

10. data handling;

11. QC measurements, including: reduction, review, confirmation, interpretation, assessment and assessment of method performance;

12. requirements specified in Section 186.185(j) of 35 IAC Part 186.

13. all information necessary to produce unequivocal , accurate records that document the laboratory activities associated with the sample receipt, preparation, analysis and reporting;

14. all information necessary to produce unequivocal link with the unique field identification and the laboratory identification code assigned each sample.

6.6 Sample Processing for Litigation[1]

The laboratory shall follow Section 6.5 of the QAP and these minimal evidentiary chain of custody procedures when processing samples for purpose of litigation.

1. Laboratories accredited for drinking water analyses, when requested to analyze a sample for possible legal action against a public water supplier, shall use evidentiary chain of custody procedures specified in the "Manual for the Certification of Laboratories Analyzing Drinking Water."

2. The laboratory shall establish and maintain the following basic requirements for evidentiary chain of custody:

   a. The evidentiary chain of custody records shall account for an unbroken possession of the sample while it is in the laboratory's custody.

   b. The evidentiary chaoin of custody records shall include signatures of all individuals who were involved with the physically handling the samples and the time of day and calender date that the sample was physically transferred from one individual to the next individual or to and from a controlled access storage area.

   c. A minimum number of persons shall be involved in sample handling.

d. The laboratory shall limit the number of documents that are required to establish evidentiary chain of custody.

e. The evidentiray chain of custody forms shall remain with the samples during transport or shipment.

f. The laboratory shall control access to all evidentiary samples and subsamples and shall document this control as dscribed in 35 IAC 186.185(j).

g. Transfer of smaples, subsamples, and digestates or extracts to another laboratory is subject to all the requirements for evidentiary chain of custody.

h. The laboratorty shall ensure that the sample containers which are shipped are sealed in such a manner so that tampering by unauthorized personnel is immediately evident.

i. The laboratory shall ensure that, if required, individual sample containers shall be sealed in such a way to prevent tampering.

j. The laboratory shall ensure that mailed packages of samples be registered with return receipt requested. If such packages are sent by commom carrier, receipts shall be retained as part of the permanent evidentiary chain of custody documentation.

6.7 Sample Disposal[1]

The laboratory maintains records for sample disposal practices, including, where appropriate, the date of sample disposal and name of responsible person.

1. If the sample is part of litigation, disposal of the physical sample will occur only with the concurrence of the affected legal authority, sample data user and submitter of the sample.

2. If the sample is subject to evidentiary chain of custody, the laboratory will document and retain a record of all conditions of disposal and all correspondence between all parties concerning the final disposition of the physical sample.

3. If the sample is subject to evidentiary chain of custody, the sample records will indicate the date of disposal, the nature of disposal (such as sample depleted, sample manifested to a hazardous waste facility, sample returned to client), and identity of the individual who performed the task.

4. The laboratory has waste collection, storage, recycling and disposal procedures and policies as part of our SOP **Sample Disposal**. Where disposal practices are included as part of an approved test method, the laboratory strictly follows the approved test method's disposal practices.

6.8 Custodian Succession

In the absence of the sample custodian, the laboratory director is designated to act as custodian.

Chain of Custody Record

1265 Capital Airport Drive, Springfield, IL 62707-8490 Phone (217) 753-1148 Facsimile (217) 753-1152

Prairie Analytical Systems, INCORPORATED

Client		Client Project	
Address		Project Location	
City, State Zip Code		Sampler(s) / Phone No.	/
Phone Number		TAT / Date Required	Standard [ ] Rush [ ] /
Facsimile Number		P.O. # / Invoice To	

Sample Description (10 Characters Only)	Sampling Date	Sampling Time	Container Size	Container Quantity	*M / **P Code	Analysis and / or Method Requested	PAS Sample Number Accepted / Rejected
					/		A[ ]R[ ]
					/		A[ ]R[ ]
					/		A[ ]R[ ]
					/		A[ ]R[ ]
					/		A[ ]R[ ]
					/		A[ ]R[ ]
					/		A[ ]R[ ]
					/		A[ ]R[ ]
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					/		A[ ]R[ ]
					/		A[ ]R[ ]

* M = Matrix Code	A - Aqueous	DW - Drinking Water	NA - Non-aqueous Liquid	SE - Saline Water	S - Solids	O - Other (Specify)
** P = Preservative Code	A - None	B - HNO_3	C - H_2SO_4	D - NaOH	E - HCl	O - Other (Specify)

Relinquished By	Date	Time	Received By	Date	Time	Method of Shipment

Special Instructions:	PAS Project Code

PAS-COC 186.185 (b)

Copies: White - Client Yellow - PAS, Inc. Pink - Sampler

Sample(s) Log-In Sheet

Prairie Analytical Systems, Incorporated

Sample Custodian ___________

PAS	Collection	Laboratory Receipt		Client	Analysis and Method	Temp. °C	Accepted	PAS
Sample Number	Date	Date	Time	Sample Description	Requested	Matrix Code	Rejected	Project Code
						/	A[ ] R[ ]	
						/	A[ ] R[ ]	
						/	A[ ] R[ ]	
						/	A[ ] R[ ]	
						/	A[ ] R[ ]	
						/	A[ ] R[ ]	
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						/	A[ ] R[ ]	

Comments:

Intralaboratory Transfer Sheet

Prairie Analytical Systems, Incorporated

Sample Custodian ___________

PAS Sample Number	Container Decsription	Collection Date	Expiration Date	Sample In Date / Initials	Sample Out Date / Initials	Sample In Date / Initials	Sample Out Date / Initials	Extract In Date / Initials	Extract Out Date / Initials	Sample Disposal General or Special Waste	Diposal Date / Initials
										G [ ] SW [ ] Depleted [ ]	
										G [ ] SW [ ] Depleted [ ]	
										G [ ] SW [ ] Depleted [ ]	
										G [ ] SW [ ] Depleted [ ]	
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Note: All samples shall be disposed in accordance with the Standard Operting Procedure Sample Disposal.

Comments:

7.0 Quality Assurance Objectives

Quality assurance objectives for precision, accuracy, representation, completeness, and comparability are discussed in this section for all measurement data. Methods and specificity are described in Sections 8.0 and 9.0.

7.1 Definition of terms[1,2]

1. Acceptance limits - the data quality limits specified for analytical method performance.

2. Accreditation - the issuance by the Agency of certificates of competency to laboratories meeting the minimum standards established in 35 Illinois Administrative Code Part 186(hereafter known as Part). Accreditation is not a guarantee of the validity of the data generated by the accredited laboratory.

3. Accredited laboratory - a laboratory that has met the criteria established by this Part.

4. Accrediting authority - the state or federal agency having the responsibility and accountability to grant accreditation to laboratories.

5. Accuracy - a measure of the degree of agreement between an observed value generated by a specific procedure and a true value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations.

6. Act - the Illinois Environmental Protection Act [415 ILCS 5].

7. Agency - the Illinois Environmental Protection Agency.

8. ASTM - the American Society for Testing and Materials, West Conshohocken, PA, a not-for- profit, voluntary standards development system.

9. Analyte - a chemical element, chemical compound, or physical property.

10. Analyte of interest - the chemical element, chemical compound, or physical property for which the laboratory is performing an analysis to determine the quantity in a sample for reporting pursuant to this Part.

11. Analyzed reagents (AR) - chemicals analyzed for impurities where the level of impurities is reported in accordance with the specifications of the Committee on Analytical Reagents of the American Chemical Society.

12. Analytical standards - a solution of a compound or a mixture of compounds of known purity in an appropriate solvent used to prepare calibration standards. An analytical standard may be traceable to NIST standard reference materials.

13. Approved performance evaluation program - a performance evaluation program which meets the requirements of Section 186.175 of this Part.

14. Approved test methods - the analytical methods specified in Section 186.180 of this Part.

15. Audit - a thorough, systematic, qualitative examination of a laboratory for compliance with this Part, including but not limited to an examination of any of the following: facilities, equipment, personnel, training, procedures, documentation, record keeping, data verification, data validation, data management, data reporting, or any aspect of the laboratory's activities which affect the laboratory's ability to meet the Agency's conditions

for accreditation or comply with this Part.

16. Batch - one to 20 samples of the same matrix that are prepared together with the same process and personnel, using the same lot of reagents with a maximum time between the start of the processing of the first sample and the start of processing of the last sample being 24 hours.

17. Bias - the systematic or persistent distortion of a measurement system which causes errors in one direction (the expected sample measurement is different from the true value).

18. Blind sample - a subsample for analysis with a composition known to the submitter that is used to test the analyst's, analyst in training's, or technician's proficiency in the execution of the measurement system. The analyst, analyst in training, or technician may know the identity of the sample but not its composition. The laboratory management may know the identity and composition of the blind sample.

19. Calibrate - initial calibration.

20. Calibration Blank (CB) - a volume of distilled or deionized water containing the same reagents, solvents, acids, or preservatives contained in the calibration standards. The calibration blank is used to determine the response of the instrument to the zero concentration of an analyte of interest.

21. Calibration standard - a solution of an analyte or mixture of analytes of known purity in an appropriate solvent used to calibrate the analytical instrument response with the respect to analyte concentration.

22. Certificate (certificate of approval) - a document issued by the Agency to a laboratory that has met the criteria and conditions for accreditation as set forth in this Part. The certificate may be used as proof of accredited status. A certificate is always accompanied with a scope of accreditation.

23. Certification - accreditation.

24. Certified laboratory - an accredited laboratory.

25. Chromatographic range - the time frame over which analytes move out of the chromatography column.

26. Competence - the ability of a laboratory to meet the Agency's conditions for accreditation and to conform to the criteria contained in this Part.

27. Confidence interval - that range of values, calculated from an estimate of the mean and standard deviation, which is expected to include the population mean with a stated level of certainty.

28. Continuing calibration verification (CCV) check - the analysis of a continuing calibration verification check standard to determine the state of calibration of an instrument between recalibrations, as required by section 186.155 of this Part.

29. Continuing calibration verification check standard - a solution of an analyte or mixture of analytes of known purity in an appropriate solvent used to perform the continuing calibration verification check. The source of the analyte may be the same as the source of the calibration standard's source or it may be a second source.

30. Controlled access storage - a refrigerator, cooler, rooms or building in which samples

are held and from which samples may be removed only by authorized laboratory personnel.

31. Corrective action - an action taken by the laboratory to eliminate or correct the causes of an existing nonconformance in order to prevent the recurrence of the nonconformance.

32. Corrective action plan - a plan of corrective actions.

33. Correlation coefficient - used to measure the acceptability of initial calibration curves.

$$R = \frac{n(\sum xy) - (\sum x)(\sum y)}{\sqrt{[n(\sum x^2) - (\sum x)^2][n(\sum y^2) - (\sum y)^2]}}$$

34. Deficiency - a failure of a laboratory to meet any requirement of this part.

35. Document - any written or pictorial information describing, defining, specifying, reporting, or certifying any activities, requirements, procedures, or results.

36. Drinking water - water used or intended for use as potable water.

37. Environmental samples - samples, excluding any laboratory generated quality control samples such as matrix spikes, duplicates, and laboratory control samples, for which the laboratory analytical results will be reported pursuant to this Part.

38. Evidentiary chain of custody - the procedures and records which ensure that an intact, contiguous written record tracing the possession and handling of samples from the point that a clean sample containers are provided by the laboratory or the point of sample collection through disposal are maintained.

39. Field blank - a sample of laboratory pure water which is filled in the field during the field sampling. The field blanks are then transported to the laboratory with the field samples for analysis.

40. Field duplicate - two separate samples collected from the same source into separate containers and analyzed independently. Field duplicates are used to assess the precision of field sampling.

41. Initial calibration (ICC) - the analyses of calibration standards for a series of different specified concentrations of an analyte of interest used to define the linearity and dynamic range of the response of the instrument of an analyte.

42. Initial Calibration Verification (ICV) - the analysis of an initial calibration verification check standard to determine the state of calibration of an instrument before the sample analysis is initiated, as required by Section 186.155 of this Part.

43. Initial calibration verification standard - a solution of an analyte or mixture of analytes of known purity in an appropriate solvent used to perform the initial calibration verification.

44. Initial demonstration of method performance (IDMP) study - the procedures performed by an analyst that insures that the analyst does not analyze unknown samples via a new or unfamiliar method prior to obtaining experience as described in Section 186.160 of this Part.

45. Inorganic - all parameters not included in organic parameters.

46. Instrument detection limit (IDL) - a detection limit which is determined statistically, defined as three times the standard deviation obtained for the analysis of a standard solution at a concentration of three to five times the estimated detection limit on three consecutive days with seven consecutive measurements per day.

47. Internal Standard - an organic compound which is similar to the analytes of interest in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples. Added to the analytical sample just prior to instrument analysis and used for the basis of quantitation.

48. Laboratory - a facility that is equipped and used for the testing of samples for the fields of testing described in Section 186.160 of this Part and the approved test methods specified in Section 186.180 of this Part. A laboratory with a main facility and an annex in the same city as the main facility and within five miles of the main facility may be considered one laboratory.

49. Laboratory control sample (LCS) - an uncontaminated sample matrix with known quantities of analytes. The analytes shall be obtained from second source. The laboratory control sample is analyzed exactly like a sample to determine wether the measurement system is performing as expected using the evaluation procedures described in 186.160 of this Part and to determine wether the laboratory is capable of making accurate and unbiased measurements.

50. Least precise step - the part of the analytical procedure that results in the greatest error in measurement.

51. Linear dynamic range - the range of concentrations over which the analytical system exhibits a linear relationship between the amount of material introduced into the instrument and the instrument's response.

52. Litigation sample - a sample, knowingly analyzed by the laboratory, for possible legal action.

53. Major remodeling - any remodeling of the facility that requires a local building permit.

54. Matrix - the predominant material of which the sample to be analyzed is composed. Sample matrices are:

Aqueous (A) - any sample other than drinking water, potable water, or saline or estuarine water;

Drinking Water (DW) - water used or intended for use as potable water;

Non-aqueous liquid (NA) - organic fluid with <15% settleable solids;

Saline or estuarine waters (SE) - any aqueous sample from an ocean or estuary;

Solids (S) - soils, sediments, sludges and other matrices with >15% settleable solids: or

Chemical Waste (CW) - a product or by-product of an industrial process that results in a matrix not previously defined.

55. Matrix Spike (MS) - an aliquot of matrix fortified (spiked) with known quantities of specific analytes and subjected to the entire analytical procedure in order to determine the effect of the matrix on an approved test method's recovery system.

56. Matrix spike duplicate (MSD) - a replicate matrix spike that is prepared and analyzed in order to determine the precision of the approved test method.

57. Method blank (MB) - a sample which does not contain an analyte of interest above an acceptable level pursuant to section 186.160 and which is processed simultaneously with and under the same conditions as samples being analyzed for analytes of interest.

58. Method Detection Limit (MDL) - the minimum concentration of a substance that can be measured and reported within 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix type containing the analyte. Unless specified by the approved test method, the method detection limit shall be determined using the procedures specified in Section 186.160 of this Part.

59. Megohm-cm - megohm-centimeter.

60. mg - milligram.

61. umhos/cm - micromhos per centimeter.

62. Neat compound - an undiluted compound.

63. NIST - the United States Department of Commerce, Technology Administration, National Institute of Standards and Technology (formerly the National Bureau of Standards).

64. Operating condition - the state of the measurement system when samples are analyzed.

65. Organic - all analytes analyzed by all forms of gas chromatography and high pressure liquid chromatography (excluding ion chromatography).

66. Parameter - an analyte.

67. Pattern of peak profile recognition for identification - a series of chromatographic peaks used to identify multi-component analytes such as the aroclors, petroleum products, toxaphene, and technical chlordane. The series of peaks used to identify a multi-component analyte have characteristic sizes, shapes, and retention times.

68. PE - performance evaluation.

69. Percent recovery - used to measure accuracy and calculated as follows:

$$\%Recovery = \frac{(Spike\,Sample\,Result - Sample\,Result) \times 100}{Spike\,Added}$$

70. Percent relative standard deviation -

$$\%RSD = \frac{SD}{X} \times 100$$

$$S = \frac{\sqrt{x^2 - (\sum x)^2/n}}{N-1} \qquad = \frac{\sqrt{(x_i - x_{ave})^2}}{N-1}$$

71. Performance Evaluation program - the aggregate of providing rigorously controlled and standardized samples to a laboratory for analysis, reporting of results, statistical evaluation of the results in comparison to peer laboratories and the collective demographics and results summary of all participating laboratories.

72. Performance evaluation sample - a sample prepared by the Agency or an Agency approved performance evaluation program, whose composition is unknown to the laboratory management, analyst, analyst in training, and technician. The performance evaluation sample is provided to test wether the laboratory can produce analytical results within specified performance limits.

73. Performance evaluation testing - the determination of laboratory performance by means of comparing and evaluating tests on the same or similar items or materials by two or more laboratories in accordance with predetermined conditions.

74. Performance evaluation study - a single testing event within a performance evaluation program.

75. Plan of corrective action - a report, including specific items addressed and specific dates of completion, generated by the laboratory in response to an Agency issued notification of nonconformance with this Part.

76. Practical quantitation limit (PQL or RL) - the lowest level of measurement and reporting that can be achieved within the specified limits of precision and accuracy during routine operating conditions. Often this value is taken from the analytical method as a multiple of the MDL.

77. Precision - the measure of mutual agreement among individual measurements of a sample, usually under prescribed similar conditions, usually expressed as the standard deviation, variance, or range, either in absolute or relative terms.

78. Quality assurance - an integrated system of activities involving planning, quality control, quality assessment, reporting, and quality improvement to ensure that a product or service meets the requirements of this Part.

79. Quality assurance plan (QAP) - a written description of the laboratory's integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

80. Quality control - the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of the users.

81. Quality control acceptance limits - the statistically determined or approved test method specified limits within which a single measurements, quality control data point, series of measurements or series of quality control data points will fall when the analytical process is producing data of satisfactory quality.

82. Quality control check sample (QCS) - an aliquot of method blank fortified with a solution of the analytes of interest of known concentration obtained from an outside source. The quality control check sample is used to check either the laboratory or instrument performance.

83. Quantitating - the arithmetic process of determining the amount of analyte in a sample.

84. Relative percent difference - for duplicates A and B, RPD is calculated as follows:

$$RPD = \frac{(A - B)}{1/2(A + B)} x\ 100$$

85. Relative response factor (RF) - measure of the relative response of an analyte compared to that of its internal standard. Relative response factors are determined by analysis of calibration standards and are used in the quantitation of analytes in samples. RF's are calculated as below:

$$RRF = \frac{Ax}{Ais} x \frac{Cis}{Cx}$$

86. Replicate - two or more equal aliquots taken from the same sample container and analyzed independently for the same constituent.

87. Sample - any solution or media introduced into an analytical instrument on which an analysis is performed excluding calibration standards, initial calibration verification check standards, calibration blanks, and continuing calibration verification check standards.

88. Sample tracking - an unbroken trail of accountability that ensures the physical security of samples, data, and records.

89. Sample duplicate - a replicate.

90. Second source - a different vendor or manufacturer, or different lots from the same vendor or manufacturer.

91. Spike concentration - a specified amount of an analyte of interest in a matrix spike, laboratory control sample, or quality control check sample.

92. Standard operating procedures (SOP) - a written, laboratory specific document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

93. Statistical outlier test - a mathematical process for determining that an observation is unusually large or small relative to the other values in a data set.

94. Surrogate - an organic compound which is similar to the analytes of interest in chemical composition and behavior in the analytical process, but which is not normally found in environmental samples.

95. Standard Methods - Standard Methods for the Examination of Water and Wastes, 19th edition, 1995.

96. Traceability - the accepted or actual value of the quantity being measured.

97. True value (TV) - accepted or actual value of the quantity being measured.

98. USEPA - United States Environmental Protection Agency.

99. Validation - confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled. Validation is the process of examining a sample result to determine conformance with user's needs.

100. Verification - confirmation by examination of and provision of objective evidence that specified requirements have been fulfilled. Verification is the process of examining a result of a given activity to determine conformance with this Part.

7.2 Precision and Accuracy

There are two criteria for which quantitative limits have been established for acceptance criteria, namely, precision and accuracy. Methods to determine the level of precision and accuracy are described in Section 8.0.

The analysis of sample duplicates and matrix spike duplicates determines the analytical precision. Replicate measurements in the field determine the precision for field measurements. Sampling precision is assessed by collection and analysis of field duplicate samples.

Accuracy is determined by the analysis of blank samples, as well as spiked samples, known standards , and calibration verifications. The use of method blanks, laboratory control samples, reference standards, calibration verifications, matrix spikes, and interference check standards provides the assessment for analytical accuracy. Surrogate standards are analyzed with all samples for organic analysis.

Acceptance limits: The following limits are established as goals for measurement of Precision and Accuracy. Approved Method(s) may have more stringent QC Limits and in that case would be followed.

Parameter	Precision - RED	Accuracy - %R
Trace Metals	+/- 20%	100% +/- 25%
Inorganics	+/- 20%	100% +/- 25%
Total Organic Carbon	+/- 20%	100% +/- 25%
Total Organic Halogen	+/- 20%	100% +/- 25%
VOA's	(A)	(A)
Base/Neutrals	(A)	(A)
Acids	(A)	(A)
Pesticides/PCB's	(A)	(A)

(A) Matrix Spike/Matrix Spike Duplicates are performed on all organic analysis in a frequency of 5% of all samples processed. Acceptance criteria for these measurements are advisory only and have no bearing on sample re-analysis. Surrogate spiking is performed with each sample, and recoveries are used as criteria for data acceptability. Surrogate recoveries for each sample are shown on all analysis reports.

7.3 Representativeness, Completeness, and Comparability

Representativeness, completeness, and comparability are qualitative criteria, not quantitative measures, but play a significant role in the QAP of a laboratory operation. These elements are also discussed in this section

Representativeness expresses the degree to which the data obtained accurately represents a given sample source and is primarily dependent on the design of the sampling plan. This designed sampling plan is site or project specific to accurately reflect and represent the environmental conditions and/or the parameter variations associated with that site or project.

Completeness is also dependent upon the designed sampling plan. The goal for completeness is to insure that all data necessary for evaluation and decision making is generated.

The consistent use of sampling procedures, the analytical methods, the data reduction, and the data reporting will assure comparability of analytical results.

8.0 Internal Quality Control Procedures

The quality control requirements outlined in the following sections for elements, inorganics, and organics are general laboratory requirements. Some methods may require more detailed quality control procedures not included herein. Individual standard operating procedures should be referred to for specific method requirements.

8.1 Calibration[1]

1. The laboratory performs an initial calibration of all instrumentation and equipment as specified in the approved test method. The laboratory uses calibration standards traceable to national standards, where available.

2. If the approved test method specifies the generation of an initial calibration curve but does not specify the appropriate number of standards for use in the initial calibration curve, the laboratory establishes the appropriate number of standards for use in the initial calibration curve using the following procedure:

a. Determine a percent relative standard deviation (%RSD) of:

1.the analyses of a minimum of seven replicate measurements of a standard with a concentration at one to three times the MDL; or

2.the response factors (internal standard calibration) or calibration factors (external standard calibration) of at least three standards having concentrations that cover the expected calibration range.

b. Determine the minimum number of calibration standards to be used in the initial calibration curve by correlating the %RSD defined in subsection (2)(a) with the number of required calibration standards. The %RSD and correlating number of calibration standards are:

%RSD	Number of Calibration Standards
0 - <2	1**
2 - <10	3
10 - <25	5
>25	7

**Assumes linearity through the origin (0,0). For analytes for which there is no origin (such as pH), a two point calibration curve shall be used.

c. The number of calibration standards as determined from the table in subsection (2)(b) and a blank is used to generate the initial calibration curve of the approved test method.

d. If the calibration curve generated pursuant to subsection (2)(c) is not linear as defined in subsection (5)(d) and the approved test method allows for the use of non-linear calibration curves, additional calibration standards shall be used to define the calibration.

3. If the approved test method specifies the generation and use of a calibration curve, all sample results are reported from sample analyses within the range of the calibration curve, except when the approved test method specifically allows otherwise (for example ICP analyses above the highest calibration standard concentration but within the linear dynamic range as established by the laboratory pursuant to the applicable approved test method).

4. When the laboratory utilizes a single point calibration and the sample results will be used in a decision related to the determination of a non-occurrence of an analyte or a non-detect at the MDL of an analyte and the approved test method does not specify the concentration of the lowest calibration standard:

a. the concentration of the lowest calibration standard shall be at one to 15 times the MDL; or

b. the laboratory shall, at the initiation of sample analyses, analyze a calibration verification check standard at one to 15 times the MDL. The laboratory shall determine the acceptability of the analysis of the calibration verification check standard by:

1. utilizing the CCV check standards' acceptance criteria specified in the approved test method; or

2. if the approved test method does not specify a CCV acceptance criteria, the results of the calibration verification check standard analysis shall be within 15% of the true value or within the 95% confidence interval determined from a minimum of 20 analyses of the calibration verification check standards.

5. The laboratory subjects all initial calibration curves to a calibration linearity test.

a. The calibration linearity is determined by:

1. a linear regression analyses of the calibration curve;

2. determining the %RSD of the response factors (internal standard calibration); or

3. determining the %RSD of the calibration factors (external standard calibration).

b. The initial calibration curve is considered linear when:

1.the correlation coefficient from the linear regression analyses is 0.995 or greater;

2. the %RSD of the response factors is 15% or less;

3. the %RSD of the calibration factors is 30% or less; or

4. the correlation coefficient is less than 0.995 if the laboratory can demonstrate that the lower correlation coefficient produces accurate results for that analyte. When making the subsection (5)(b)(4) demonstration, the laboratory shall:

i. calculate the correlation coefficient for 20 calibration curves;

ii. calculate the mean and standard deviation of the subsection (5)(b)(4)(i) correlation coefficients;

iii. calculate the new minimal, acceptable correlation coefficient as the mean minus two standard deviations determined in subsection (5)(b)(4)(ii); and

iv. then analyze a standard prepared at a concentration which is 40% to 60% of the maximum calibration ran-e and from a second source material than that used in the calibration curve.

5. After completing the subsection (5)(b)(4) demonstration, the laboratory may consider a calibration curve linear when:

i. the correlation coefficient meets or exceeds the new criteria determined in subsection (5)(b)(4)(iii); and

ii. when the result of the subsection (5)(b)(4)(iv) analysis is within 5% of that standard's true value.

c. If the initial calibration curve is linear as determined pursuant to:

1. subsection (5)(b)(1) or (4), the laboratory shall utilize the linear regression to determine the analytical results;

2. subsection (5)(b)(2), the laboratory shall utilize the average response factor to determine the analytical results; or

3. subsection (5)(b)(3), the laboratory shall utilize the average calibration factor to determine the analytical results.

d. If the initial calibration curve is not linear as determined pursuant to subsection (5)(b), the laboratory shall utilize the entire initial calibration curve to determine analytical results.

6. To verify all initial calibration curves, the laboratory performs analyses of an initial calibration verification (ICV) check standard for all instrumentation and equipment.

a. The laboratory utilizes only ICV check standards prepared from a second source, where available.

b. The laboratory utilizes only ICV check standards prepared at the concentrations specified in the approved test method.

c. If the approved test method does not specify the concentration for the ICV check standard, the concentration is at 10% to 50% of the maximum of the calibration range.

d. The laboratory utilizes the ICV check standards' acceptance criteria specified in the approved test method.

e. If the approved test method does not specify the ICV acceptance criteria, the results of the analyses of the ICV check standard are within 15% of the true value or within the 95% confidence interval determined from a minimum of 20 analyses of the ICV check standards.

7. If the analyses of the ICV check standard fails to meet the acceptance criteria specified in subsection (6)(d) or (e), the laboratory:

a. either:

1. suspends sample analyses and take corrective action to be followed immediately by a reanalysis of the ICV check standard; or

2. immediately reanalyzes the ICV check standard; and

b. evaluate the subsection (7)(a)(1) or (2) ICV check standard reanalysis results as follows:

1. The laboratory may continue sample analyses for the analytes for which the results of the reanalysis of the ICV check standard meet the acceptance criteria specified in subsection (6)(d) or (e).

2. The laboratory terminates sample analyses or rejects sample analyses data for the analytes for which the results of the reanalysis of the ICV check standard fail to meet the acceptance criteria specified in subsection (6)(d) or (e).

3. The laboratory proceeds with sample analyses for the analytes for which the acceptance criteria were not met only after the establishment and verification of a new initial calibration curve pursuant to this section.

8. To verify the continued acceptability of the initial calibration, the laboratory prepares and performs the analysis of a CCV check standards for all instrumentation and equipment according to the following procedure:

a. The laboratory utilizes a CCV check standard prepared from the initial calibration curve standards or from a second source material than that used to prepare the initial calibration curve standards.

b. The laboratory prepares a CCV check standard at a concentration within the range of the initial calibration standards.

c. Whenever the laboratory does not prepare an initial calibration curve on the day of analysis, the laboratory shall verify the integrity of the initial calibration curve at the beginning of each day of use (or 24 hour period).

1. The laboratory initially analyzes a CCV check standard:

i. at the approved test method specified concentration, or

ii. if the approved test method does not specify the concentration for the CCV check standard, the concentration shall be at 25% to 50% of the maximum of the calibration range.

2. The laboratory analyzes a calibration blank.

3. The analysis of the-CCV check standard must meet the acceptance criteria specified in subsection (8)(d) or (e).

d. The laboratory analyzes a CCV check standard once per 20 samples or every 12 hours, whichever is more frequent.

e. The laboratory utilizes the CCV check standards' acceptance criteria specified in the approved test method.

f. If the approved test method does not specify the CCV acceptance criteria, the CCV check result are within 15% of the true value or within the 95% confidence interval determined from a minimum of 20 analyses of the CCV check standard at a single concentration.

9. If the analyses of the CCV check standard fails to meet the acceptance criteria specified in subsection (8)(d) or (e), the laboratory:

a. Either:

1. suspends sample analyses and takes corrective action followed by an immediate reanalysis of the CCV check standard; or

2. immediately reanalyzes the CCV check standard; and

b. Evaluate the subsection (9)(a)(1) or (2) CCV check standard reanalysis results as follows:

1. The laboratory may continue sample analyses for the analytes for which the results of the second analysis of the CCV check standard meet the acceptance criteria specified in subsection (8)(d) or (e).

2. The laboratory terminates sample analyses or rejects sample analyses data pursuant to subsection (10) below for the analytes for which the results of the second analysis of the CCV check standard fail to meet the acceptance criteria specified in subsection (8)(d) or (e).

3. The laboratory may proceed with sample analyses for the analytes for which the acceptance criteria were not met only after the establishment and verification of a new initial calibration curve pursuant to this Section.

10. Whenever the generation of a new initial calibration curve and verification of the new initial calibration curve are required pursuant to subsection (9), the laboratory reanalyzes all samples analyzed since the last CCV check standard which met the CCV acceptance criteria, except for those instances where the CCV acceptance criteria was exceeded high (high bias) and there are non-detect results for the corresponding, analyte in the samples associated with the CCV check standard. In those instances, the non-detect results may be reported.

11. The laboratory documents all activities related to calibration and standardization as specified in 35 IAC Part 186.190.

8.2 Quality Assurance/Quality Control[1]

1. The laboratory follows the quality control procedures specified below:

a. The laboratory follows all quality control procedures in the approved test method. The laboratory utilizes the quality control procedures set forth in this Section if the approved test method does not specify any quality control procedures or the quality control procedures contained in the approved test method are less stringent.

b. The laboratory assess's and evaluates the results of all quality control procedures, including but not limited to those procedures specified in subsections (1)(c), (d), (e), (f) and (g) on an on-going basis.

1. The laboratory establishes written procedures to ensure that all results from all quality control procedures are reviewed and the decision made to accept, reject, or qualify sample data before the data is reported.

2. The laboratory establishes written criteria for accepting, rejecting, or qualifying sample data based on each quality control procedure.

i. The laboratory, for each quality control procedure, uses the acceptance criteria contained in the approved test method for evaluating the results of each of the quality control procedures and for accepting, rejecting, and qualifying- sample data.

ii. The laboratory establishes written criteria if the approved test method does not specify the criteria for evaluating the results of each of the quality control procedures and for accepting, rejecting, and qualifying data.

3.If a quality control procedure results in the laboratory rejecting or qualifying sample data, the laboratory implements corrective actions.

4. The laboratory completes corrective actions and maintains written records as required in 35 IAC 186.190.

c. The laboratory prepares and analyzes a method blank with each batch of environmental samples and carries the method blank through the entire analytical process. Method Blanks are not required for approved test methods, including but not limited to: pH, temperature and conductivity, for which method blanks are not appropriate.

1. A batch of drinking water sample data meets the requirements of this Section only when the method blank does not contain an analyte of interest at a concentration greater than the MDL.

2. A batch of environmental sample data, except for drinking water sample data, meets the requirements of this Section when the method blank does not contain an analyte of interest at a concentration greater than the highest of the following:

i. the MDL,

ii. 10% of the regulatory limit for that analyte, or

iii. 10% of the measured concentration for that analyte in any environmental sample in the batch.

3. The provisions of subsection (1)(c)(2) do not apply in those instances where the method blank criteria have not been met and there are non-detect results for the corresponding analyte in the environmental samples associated with the method blank. In such instances, the non-detect results may be reported without a qualification.

d. The laboratory performs matrix spikes at a rate of one per 20 or fewer environmental samples per matrix type, per sample extraction or preparation procedure.

1. The laboratory utilizes the spiking analytes specified in the approved test method, except when the approved test method indicates that all method analytes are to be matrix spiked. In such cases, the laboratory spikes the analytes of interest.

2. If the approved test method does not specify the spiking analytes, the laboratory :

i. spikes 10% of the analytes listed in the approved test method, or a minimum of three analytes of interest, whichever is greater (if the approved test method lists fewer than three analytes, the laboratory shall spike all analytes of interest),

ii. spikes at least one multi-component analyte when the approved test method includes multi-component analytes (for example: chlordane, toxaphene and PCBs in USEPA Method 608), and

iii. selects analytes for spiking on a rotating, basis from among the approved test method listed analytes, for approved test methods which list more than six analytes. The laboratory shall rotate the analytes for spiking over a two-year time period, ensuring, that all analytes of interest are used in the time period. The analytes selected for spiking shall represent all chemistries, elution patterns and masses.

3. The laboratory selects samples on a rotating basis to receive matrix spike analysis from among, various client samples, waste streams, monitoring locations and other applicable locations.

4. The laboratory documents as required in 35 IAC 186.190(d)(1) the procedure used to select the sample for matrix spike analyses.

5. The laboratory documents as required in 35 IAC 186.190(d)(1) the procedure used to select the analytes for matrix spike analyses.

6. Matrix spikes are not required for approved test, methods in which materials for matrix spiking are not available, including but not limited to: total suspended solids, total dissolved solids, total volatile solids, flash point, reactivity, pH, color, odor, temperature, dissolved oxygen and turbidity.

e. The laboratory analyzes laboratory control samples (LCS) at a minimum of one per batch, except for analytes for which spiking, solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity.

1. The laboratory uses the results of these LCS analyses to determine batch acceptance.

2.The laboratory often uses the matrix spike samples as specified in subsection (1)(d) as an LCS when the matrix spike acceptance criteria are as stringent as the LCS acceptance criteria. However, if the laboratory prepares an LCS, the laboratory analyzes the LCS and uses the results to determine batch acceptance. The laboratory does not use the analyses of matrix spike samples as specified in subsection (1)(d) to override, ignore, or replace an LCS analysis that fails to meet criteria.

3. The analytes are obtained from a second source, if applicable.

f. The laboratory performs matrix spike duplicates or sample duplicates at a rate of one per 20 or fewer environmental samples per matrix type, per sample extraction or preparation procedure.

1. The laboratory performs matrix spike duplicates on the same environmental sample chosen for matrix spike analyses pursuant to subsection (a)(4)(C).

2. The laboratory selects samples on a rotating, basis to receive sample duplicate analyses from among various client samples, waste streams, monitoring locations and other applicable locations.

3. The laboratory documents, as required in 35 IAC 186.190(d)(l 1), the procedures used to select the sample for matrix spike duplicate or sample duplicate analyses.

g. The laboratory adds surrogate compounds to all samples, standards, and blanks, whenever possible, when conducting analyses by approved test methods utilizing organic chromatography.

h. The laboratory maintains tabulations, quality control charts and any combination of tabulations and quality control charts of the results from all quality control procedures, excluding blanks, which have criteria established pursuant to subsection (1)(d) above:

1. for each approved test method;

2. for each matrix; and

3.for each analytical range. The laboratory calculates quality control limits according to Standard Methods Part 102OB(7)(a) and (b) or AOAC "Quality Assurance Principles for Analytical Laboratories."

i. Tabulations, quality control charts or any combination of tabulations and quality control charts of results of quality control procedures shall include the following information:

1. title;

2. identification of standard operating procedure (SOP) which requires collection of quality control procedure data;

3. name of quality control procedure being tabulated;

4. analytical method;

5. analyte;

6. analyte units of measure;

7. matrix;

8. fortification concentration;

9. mean;

10. standard deviation;

11. upper control limit (UCL);

12. lower control limit (LCL);

13. upper warning limit;

14. lower warning limit (LWL);

15. date of analyses;

16. unique control sample identification code; and

17. analyst's identification.

j. Each analyst performs an IDMP study prior to initiation of sample analyses, unless the IDMP is not applicable to the approved test method, such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The laboratory is responsible for the repetition of the IDMP study whenever there is a change in analyst, instrument type, or approved test method. The following steps are performed:

1. A quality control (QC) check sample is obtained from USEPA or a certified source. If not available, the QC check sample is prepared by the laboratory using calibration standards that are prepared at a different time than those used in instrument calibration.

2. The laboratory prepares four aliquots of the QC check sample at the required method volume to a concentration approximately 10 times the method-stated or laboratory-calculated MDL.

3. The four aliquots are prepared and analyzed according, to the approved test method.

4. Using the four results, calculate the average recovery in the appropriate reporting units (such as µg/L) and the standard deviation (in the same units) for each analyte.

5. For each analyte, compare standard deviation and average recovery to the corresponding acceptance criteria for precision and accuracy in the approved test method (if applicable) or laboratory-generated acceptance criteria (if a non-standard method). If standard deviation and average recovery for all analytes meet the acceptance criteria, the

analysis of actual samples may begin. If any one of the analytes exceed the acceptance range, the performance is unacceptable for that analyte.

6. When the results of the IDMP indicates that the average recovery or the standard deviation of one or more of the tested analytes does not meet the acceptance criteria pursuant to subsection (1)(j)(5), the analyst:

i. locates and corrects the source of the problem and repeats that portion of the IDMP specified in subsections (1)(j)(3), (4) and (5) for applicable analytes; or

ii. repeats that portion of the IDMP specified in subsections (1)(j)(3), (4) and (5) for applicable analytes. If the results of the IDMP conducted pursuant to this subsection (1)(j)(6)(ii) fails to meet the acceptance criteria, the Agency will deem a general problem with the measurements system to exist. The analyst must then follow the requirements of subsection (1)(j)(6)(I).

7. The laboratory provides the Agency with the information as specified in the application process, 35 IAC 186.125(d)(15)(C).

k. The laboratory determines MDLs using the procedures specified in 40 CFR 136 Appendix B, unless the approved test method specifies the procedure for MDL determination or the determination of an MDL is not applicable to the approved test method, such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity.

1. The laboratory analyzes seven replicates to determine the MDL.

i. The laboratory uses all analytical results when calculating the MDL.

ii. If the laboratory analyzes more than seven replicates, the laboratory shall only exclude analytical results which the laboratory determines are outliers by utilizing a statistical outlier test. Statistical outlier tests include, but are not limited to: The Rule of Huge Error, Dixon Test for Outlying Observations, and Grubbs Test for Outlying Observations, as set forth in "Quality Assurance for Chemical Measurements."

2. The calculation of MDLs pursuant to 40 CFR 136 Appendix B procedures may not be appropriate for multi-component analyses such as aroclors, toxaphene, and technical chlordane because they require a pattern of peak profile recognition for identification. The laboratory shall define the MDL for multi-component analyses as the lowest concentration for which pattern recognition is possible.

3. The laboratory determines MDLs for each approved test method:

i. annually; and

ii. when there is a change in instrument type.

4. The laboratory, in lieu of the annual determination of the MDL pursuant to subsection (1)(k)(3), annually verifies the MDL by the preparation and analysis of a minimum of one matrix spike sample, spiked at the current MDL.

i. An MDL is considered verified and acceptable for continued use if the results of the analysis of the clean matrix spike sample is within the 95% confidence interval as set forth in 40 CFR 136 Appendix B.

ii. If an MDL cannot be verified pursuant to subsection (1)(k)(4)(I), a new MDL shall be determined.

5. The laboratory provides the Agency with all of the MDL information as specified in the application process, 35 IAC 186.125(d)(I5) and (I 7).

6. The laboratory uses +/- 50% of the true value of the MDL concentration for replicate percent recovery acceptance.

2. An MDL calculated pursuant to the requirements of this Section is valid when:

a. Thee calculated MDL is greater than 1/10 the MDL spiking concentration;

b. The MDL spiking concentration is greater than the calculated MDL;

c. The laboratory has met its criteria for acceptable replicate percent recovery; and

d. For drinking water laboratory accreditation, the laboratory has achieved MDLs equal to or less than those specified in Appendix A of this Part for all analytes listed for the approved test method.

3. The laboratory repeats the MDL study if the criteria specified in subsection (b) are not met.

4. The laboratory arranges for and has conducted annual internal audits of the technical activities to verify that its operations or procedures continue to comply with this Section.

a. Such internal audits are performed by the quality assurance officer or designee who is trained and qualified as an auditor and who is, wherever possible, independent of the activity or procedure audited.

b. Where the results of the internal audit indicate that operations or procedures are not in compliance with this Part, corrective action shall be taken pursuant to 35 IAC 186.165.

c. Where results of the internal audit indicate that the laboratory's test results are invalid, the laboratory shall take immediate corrective action and shall immediately notify, in writing,, any clients whose data are affected.

8.3 Laboratory Water Quality Control

Laboratory pure water is generated from the tap water provided by the City of Springfield and is then fed through a Barnstead water purification system. The quality of the finished lab water is monitored by a resistance meter located at the outlet of the Barnstead unit. Resistance must be > 15 megohm-cm. Readings less than the stated criteria indicate the need to replace the filters in the system.

8.4 Sample Bottle Quality Control

One container from each lot of 50 bottles shall be tested for contamination. One exception is in the case of VOA vials, where one in every 25 vials shall be tested. Container lots are tracked by the use of a date stamp, with which every cleaned container is labeled and dated.

Acceptance criteria are:

1.demonstration that the minimum required numbers of containers are tested for contamination, and

2. that levels of contamination do not exceed the detection limits of analytes for which the container is to be used.

Contamination above the detection levels will result in the rejection of the entire lot of containers. The lot shall be recleaned and reanalyzed until QC criteria are met. For analysis on all containers, concentrations are calculated on a full-bottle basis. For some analysis, however, the container may not be filled to capacity.

Each container selected for quality control analysis is logged in and processed in the same manner as all other samples. All results are kept on file in the laboratory.

Analyses are conducted utilizing EPA and other accepted methodology. GC-MS analysis used for the detection of all organic contaminants is not required.

Sample collection procedures are as follows:

1. Extractable Organic Compounds -1/2 gallon amber glass 8 oz. glass (solids)

Add 60 ml pesticide grade methylene chloride, cap securely and shake well. Store at 4°C until analysis.

2. Purgeable Organic Compounds - 40 ml glass.

Fill with lab pure water and cap, allowing no headspace. Store at 4°C until analysis.

3. Metals - 16 oz. and 32 oz. HDPE.

Fill container to capacity with lab pure water. Add 1 ml HNO_3, cap and shake well. Store at 4°C until analysis.

4. Cyanide - 32 oz. HDPE.

Fill container to capacity with lab pure water. Preserve to pH>12 with NaOH, cap and shake well. Store at 4°C until analysis.

5. Phenol, TOX, TOC - 32 oz. amber glass.
8 oz. amber glass.

Fill container to capacity with lab pure water. Preserve to pH<2 with H_2SO_4, cap and shake well. Store at 4°C until analysis.

6. Oil and Grease - 32 oz. glass

Fill container with lab pure water. Preserve to pH<2 with H_2SO_4, cap and shake well. Store at 4°C until analysis.

7. Ammonia, Phosphorous, TKN, COD, Organic Nitrogen, Nitrate, Nitrite - 32 oz. HDPE.

Fill container with pure lab water. Preserve to pH <2 with H_2SO_4, cap and shake well. Store at 4°C until analysis.

8.All other unpreserved parameters - ½ gal. HPDE = High Density Polyethylene Container.

Fill container with pure lab water, cap and shake well. Store at 4°C until analysis.

All analyses shall be conducted using the appropriate operating procedure. All analyses shall be accompanied by routine procedural QC practices. Appropriate hold times shall be observed.

9.0 Analytical Methodology

9.1 Analytical Methods

There is a wide variety of analytical methods utilized by Prairie Analytical Systems, Inc., which are approved by the various regulatory agencies. The sample type and the final use of the data by the client mandates the method of choice. Methodology may also be determined by the contract or the conditions of certification.

Modification of standard analytical procedures, in many cases, may result in improved method performance or in increased sensitivity. For most of the methodology used at this facility, the procedures are derived from the following sources:

1. **"Test Methods for Evaluation of Solid Waste, SW846" , "Laboratory Manual Physical/Chemical Properties"**, Volume 1A, 1B and 1C, 3rd edition, Office of Solid Waste and Emergency Response, Environmental Protection Agency.

2. **"Standard Methods for the Examination of Water and Wastewater"**, 19th Edition (1995), American Public Health Association, American Water Works Association, Water Pollution Control Federation, 1995.

3. EPA No. 600/4-82-057, **"Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater"** (March 1982) U. S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory.

4. EPA No. 600/4-79-020, **"Methods for Chemical Analysis of Water and Wastes"** (March 1983) U. S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory.

5. Code of Federal Regulations 40 CFR Office of the Federal Register:

   40 CFR 136.4 (1997),
   40 CFR 136.5(1997),
   40 CFR 136 Appendix A (1997),
   40 CFR 136 Appendix B (1997),
   40 CFR 136 Appendix C (1997),
   40 CFR 141.23(k) (1997),
   40 CFR 141.24(e) (1997),
   40 CFR 141.27 (1997), and
   40 CFR 143.4 (1997).

6. EPA No. 815-B-97-001, **"Manual for the Certification of Laboratories Analyzing Drinking Water"** 4th edition, March 1997. U.S. Environmental Protection Agency, Office of Water, Office of Groundwater and Drinking Water, Technical Support Center, Ohio 45268)

7. EPA No. 821-C-97-001, **" Methods and Guidance for Analysis of Water"** Version 1.0, April 1997. U.S. Environmental Protection Agency, Analytical Methods Staff, Engineering and Analysis Division, Washington, DC 20460.

8. **Methods OA-1 & OA-2**, UST Program, Environmental Protection Division, Iowa Department of Natural Resources, Des Moines, IA. Rev. July 1991

9.2 Departures from Analytical Methods

Alternate methods developed internally must be demonstrated to be of equal or greater performance than the existing method referenced in 40 CFR Part 261, 264 or 265 and published in SW 846. The following requirements must be achieved before an alternate test method is used:

1. A complete procedure for the test method must be written, including all equipment used for the method.

2. The matrices for which the method will be used must be described.

3. Comparative results of the proposed method with the existing method must be given that display supportive information for the proposed test.

4. Some assessment of the interferences which may prohibit the proposed test method must be included.

5. The quality control practices to monitor the proposed test method must be described.

This information will be reviewed by the Laboratory Director and the Quality Assurance Officer and if acceptable, will be formally submitted to the USEPA for petition as an alternative test method.

9.3 Standard Operating Procedures[1]

The laboratory maintains for each approved test method written, laboratory specific Standard Operating Procedures (SOP's) that accurately reflect all phases of current laboratory practices such as assessing data integrity and corrective actions. Copies of the Standard Operating Procedures (SOP's) used by this facility are available upon request. In making a SOP the following topics, where applicable, should be addessed:

1. Scope and application. This topic includes the list of analytes, the matrices to which the approved test method applies, a generic description of method sensitivity, and a description of method limitations. Much of this information is presented in tabular format.

2. Summary of approved test method. This topic summarizes the approved test method in a few paragraphs. The purpose of the summary is to provide a succinct overview of the technique to aid the reviewer or data user in evaluating the approved test method and the data. List sample volume, extraction, digestion, concentration, and other steps employed, the analytical instrumentation and detector systems and the techniques used for quantitative determinations.

3. Definitions. This topic includes the definitions of all method specific terms. For extensive lists of definitions, refer to Section 7.1 in the Quality Assurance Plan.

4. Interferences. This topic needs to discuss any known interferences that are specific to the approved test method.

5. Safety. This topic needs to discuss only those safety issues specific to the approved test method and beyond the scope of routine laboratory practices. Target analytes or reagents that pose specific toxicity or safety issues need to be addressed in this topic.

6. Equipment and Supplies. This topic must state the equipment and supplies that were used in performing the approved test method.

7. Reagents and Standards. This topic must provide details on the concentration and

preparation of reagents and standards to allow the work to be duplicated.

8. Sample collection, preservation and storage. This topic must provide information on sample collection, preservation, shipment and storage conditions.

9. Quality control. This topic must describe specific QC steps, including such procedures as method blanks, laboratory control samples, QC check samples and instrument checks. This topic must define all terms not previously defined pursuant to 3. This section must include the frequencies for each QC operation.

10. Calibration and standardization. This topic must discuss the initial calibration process, indicate frequency of such calibration, refer to performance specifications and indicate corrective actions that must be taken when performance specifications are not met. This topic also may include discussions of procedures for calibration verification or continuing calibration, if those procedures are not included in 11.

11. Procedure. This topic must provide a general description of the sample processing and instrument analyses steps.

12. Data analysis and calculations. This topic must describe qualitative and quantitative aspects of the approved test method, list identification criteria that are used and provide the equations that are used to derive the final sample results.

13. Method Performance. This topic must provide a detailed description of the approved test method performance, including data on precision, bias, detection limits and statistical procedures used to develop performance specifications.

14. Pollution prevention. This topic must describe aspects of the analytical method that minimizes or prevents pollution.

15. Waste Management. This topic must describe the waste management practices specific to the approved test method.

16. References. This topic must site the source documents and publications, including the approved test method.

17. Tables, diagrams, flow charts and validation data. This topic must provide additional information and may be presented at the end of the approved test method. Lengthy tables may be included here and referenced elsewhere in the text by number.

Each SOP shall contain on each page the following:

1. SOP Number;

2. revision number;

3. date; and

4. current page number of total pages of a section.

10.0 Data Reduction, Validation and Reporting

10.1 General

Generation and handling of vast amounts of data require a well managed system as in integral part of the facilities QAP. The purpose of this section is to describe the handling and flow of data from the collection of raw data through the storage of validated data. The discussion includes the maintenance of logbooks and data sheets, error correction, and data reduction, transfer, validation, and reporting.

10.2 Logbooks and Data Sheets

All laboratory raw data is in the form of hand written entries onto numbered pages in data logbooks, data worksheets or computer printed data sheets for inorganic and organic analysis instrumentation.

Computerized printouts contain information relating to PAS sample number, sample description, date of analysis and analytical data. Analytical data includes the calibration data; analyte response for all samples including check standards, continuing calibration checks and analytical samples; analyte concentrations. These printouts are maintained chronologically in bound form in binders designated for each individual instrument.

For analytical methods which do not have computer printouts, all data entries and calculations are manually entered into an analysis logbooks or data worksheets. A separate logbook is maintained for each analytical parameter/test method. Information entered into these logbooks is the same for instrument printouts.

10.3 Error Correction

The analyst or section supervisor performs any correction of errors relating to sampling data. Incorrect data on instrument printouts or in manually entered data in logbooks or data worksheets are corrected in ink by lining out the original data or calculation with a single line and entering the corrected information. Corrected information shall be initialed by the individual making the correction and adding a brief explanation of the action, if appropriate.

10.4 Data Reduction

Data reduction involves the manipulation of raw sample data including detector responses, titrant volumes and gravimetric measurements to achieve final sample analyte concentrations.

The method of calculation of results from raw data are detailed in the individual analytical methods discussed herein.

10.5 Data Transfer

The analytical results for each sample are entered into the laboratory information management system which identifies each sample by a unique laboratory sample number. Each analyst enters sample results, following data reduction and validation, into the management system which stores all sample results until all required data has been entered. A sample report is issued by the Laboratory Director after all required results have been entered and validated.

10.6 Data Validation and Reporting

Data is evaluated by the analyst and/or section supervisor prior to its entry into the laboratory management system in terms of adherence to acceptance criteria for precision, accuracy, and completeness.

Data is further checked by the Laboratory Director and/or the Quality Assurance Officer prior to generating a sample report in terms of accuracy, consistency, comparability, and completeness in relation to its intended use. A signed report is submitted to the client and a copy of the signed report, along with all supporting laboratory documentation, is maintained on file at the laboratory.

1. Reporting of Significant numbers - The laboratory follows the established guidelines in the ***Handbook for Analytical Quality Control in Water and Wastewater Laboratories***, USEPA, (June 1972) when reporting data generated from the analysis of a sample. The term *"significant figure"* describes the judgement of the reporting digits in a result. The following describes the process of retention of significant figures:

a. A number is an expression of quantity. A figure or digit is any of the characters 0,1,2,3,4,5,6,7,8,9; which, alone or in combination, serve to express a number. A significant figure is a digit that denotes the amount of the quantity in a particular decimal place in which it stands. Reported analytical values should contain only significant figures. A value is made up of significant figures when it contains all digits known to be true and one last digit which is in doubt. For example, if a value is reported as 18.8 mg/l, the 18 must be firm while the 0.8 is uncertain, but, presumably better than one of the values 0.7 or 0.9 would be.

b. Final zeros after a decimal point are always meant to be significant numbers. For example, to the nearest milligram, 9.8g is reported as 9.800g.

c. Zeros before a decimal point with nonzero digits preceding them are significant. For example, in the number 209, the zero is significant. With no preceding nonzero digit, a zero before the decimal point is not significant.

d. If there are no nonzero digits preceding a decimal point, the zeros after the decimal point but preceding other nonzero digits are not significant. These zeros only indicate the position of the decimal point. As in the example, in the number 0.004, the zeros are not significant.

e. Final zeros in a whole number may or may not be significant. In a conductivity measurement of 1,000 umho/cm, there is no implication by convention that the conductivity is 1000 +/- 1 umho. Rather, the zeros only indicate the magnitude of the number.

f. A good measure of the significance of one or more zeros interspersed in a number is to determine whether the zeros can be dropped by expressing the number in exponential form. If they can, the zeros may not be significant. For example, no zeros can be dropped when expressing a weight of 100.08g in exponential form; therefore the zeros are significant. However, a weight of 0.0008g can be expressed in exponential form as 8×10^{-4}g, so the zeros are not significant. Significant figures reflect the limits of accuracy of the particular method of analysis. It must be decided whether the number of significant digits obtained for resulting values is sufficient for interpretation purposes. If not, there is little that can be done within the limits of the given laboratory operations to improve these values. If more significant figures are needed, a further improvement in method or selection of another method will be required.

g. Once the number of significant figures obtainable from a type of analysis is established, data resulting from such analyses are reduced according to set rules for rounding off.

h. Rounding off numbers - A necessary operation in all analytical areas. It is automatically applied by the limits of measurement of every instrument and all glassware. However, when it is applied in chemical calculations incorrectly or prematurely, it can adversely affect the final results. Rounding off should be applied only as described as follows:

i. If the figure following those to be retained is less than 5, the figure is dropped, and the retained figure is kept unchanged. Eg., 11.443 is rounded off to 11.44.

ii. If the figure following those to be retained is greater than 5, the figure dropped, and the last figure is raised by 1. Eg., 11.446 is rounded off to 11.45.

iii. If the figure following those to be retained is 5, and if there are no figures other that zeros beyond the five, the figure 5 is dropped, and the last place figure retained is increased by one if it is an odd number or kept unchanged if it is an even number. Eg., 11.435 is rounded off to 11.44 while 11.425 is rounded off to 11.42.

iv. The question of significant figures also arises in reading an analog instrument, i.e., analog meter, mercury-in-glass thermometer, peak heights, et cetera. Generally, all but the last digits are known with certainty. There is, however, uncertainty in the last digit. For the purposes of this facility, the limit of uncertainty will be +/- 3, that is a range of 6. If the last digit is not known within this range, one (1) significant digit should be dropped.

10.7 Report Issuance[1]

The laboratory issues sample data or sample results accurately and in a manner that is understandable to the recipient. The basic information included in the report includes the following:

1. report title, such as "Certificate of Results" or " Laboratory Results" with the accreditation number, name, address and phone number of the laboratory;

2. name and address of client and project;

3. unique identification of the report and of each page and the total number of pages;

4. description and identification of samples;

5. date of sample receipt, sample collection and sample analysis (time of sample preparation and analysis if the required holding time for either activity is less than or equal to 48 hours);

6. approved test method utilized;

7. sample results with any failures or deviations from approved test methods or QC criteria identified, such as data qualifiers;

8. signature, or name if electronic, and title of the individuals accepting responsibility for the content of the report and date of issue;

9. clear identification, including the lab name and accreditation number pursuant

to the requirements set forth in Section 35 IAC 186.195, of any samples that were gathered by a subcontract laboratory;

10. a description of the calculations or operations performed on the data, a summary and analysis of the data, and a statement of conclusions drawn from the analysis;

11. identification of the reporting units, such as µg/l or mg/kg;

12. a statement that the report shall not be reproduced, except in full, without the written approval of the laboratory, where appropriate;

13. where applicable, a statement to the effect that the sample results relate only to the analytes of interest tested or to the sample as received by the laboratory;

14. where applicable, characterization and condition of the sample;

15. where applicable, reference to the sampling procedure;

16. clear and unequivocal identification of the analytical results generated by an approved test method for which the laboratory is accredited in accordance with the laboratory's accreditation pursuant to 35 IAC 186.

11.0 Internal Laboratory Audits

There are two types of laboratory audit procedures routinely conducted by Prairie Analytical Systems, Inc., namely, performance audits and system audits.

11.1 System Audits

System audits, consisting of the evaluation of the control measurement systems and of the data management systems, are conducted annually by the Quality Assurance Officer, to ensure that all elements of the Quality Assurnace Plan are being followed.

1. Control Measurement Systems. - These systems audits include a thorough review of all documentation related to internal QC checks and the evaluation of accuracy, precision, and completeness of data. Adherence to acceptance criteria of laboratory control sample results, spike recovery, and sample duplicate results are also reviewed. Documentation is inspected to confirm that work is being conducted in accordance with special project requirements, when or if necessary.

2. Data Management Systems - All records and files are maintained as electronic and/or printed data. This system audit provides for the continual observation of sample management, including form revisions or variations, as required by the client base of the facility.

11.2 Performance Audits

The performance audit is the laboratory's method of obtaining a quantitative evaluation of the measurement systems within the facility. Performance audits, conducted periodically, consist of blind samples, prepared from certified solutions as check standards, which are logged and processed as a routine analytical sample. Performance audits are usually conducted as part of the employee training and certification processes, and as a follow-up to corrective action requests resulting from unacceptable results on an external performance evaluation study.

Results of all performance evaluation samples are reported to the Quality Assurance Officer. These results are viewed and used to determine if problems exist with a technique, a method, or an analytical system that could affect sample results.

In conjunction with the Drinking Water Certification by the Illinois Environmental Protection Agency, a biennial inspection of the facility and an audit of the lab's performance will be conducted by a representative of that Agency.

The entire laboratory will participate in two Water Supply Performance Evaluation Studies per year. This program is sponsored by and reported to the United States Environmental Protection Agency, EMSL, Cincinnati, Ohio.

12.0 <u>Corrective Action</u>

Whenever testing discrepancies are detected, or there are departures from documented policies and procedures, a corrective action policy and procedures plan is implemented. The corrective action program enables the facility to identify, document and correct any problems that may affect the quality of the analytical data, and is intended to prevent reoccurrence of similar problems in the future.

12.1. Identification of Problems

It is important to identify problems that affect the integrity and/or quality of the analytical performance of the facility. Once the problem is identified, an anticipated or recommended corrective action plan can be implemented. The more common problems that are listed below:

1. Any USEPA/IEPA PES Failure.
2. Any blind/double-blind Quality Control sample failure.
3. Calibrations that are "out-of-control".
4. Any bottle Quality Control failure.
5. Chronic sample/procedural blank contamination.
6. Any noted malfunctions of instruments or equipment.
7. Any monthly balance calibration criteria failure.
8. Chronic Lab Control failure.
9. Any Control Sample failure.

The anticipated or recommended corrective actions are routinely conducted as they are needed and are performed in accordance with the following procedures.

12.2 Individuals Responsible for Initiating Corrective Action

1. <u>Method Corrective Action</u> - The analyst or the section supervisor would initiate a Method corrective action on a daily or on a "as needed" basis. A Method corrective action may also be initiated as a result of not meeting acceptance criteria for internal QC checks, including poor recovery, precision, or instrument response.

2. <u>System Corrective Action</u> - The Quality Assurance Officer initiates the System corrective action procedure by a written request to the section supervisor and/or analyst. This type of action is usually initiated due to poor performance audit results, poor system audit results or unacceptable results for a PES by a regulatory agency.

12.3 Individuals Responsible for Investigating the Problems Identified.

1. <u>Method Corrective Action</u> - When corrective action is required, analysis must be stopped until the problem has been identified and corrected. When QC checks verify that the problem has been identified and corrected, analysis may continue. Samples analyzed after the last acceptable QC check must be reanalyzed. Documentation describing the source of the problem and the actions taken to correct the problem must be entered into the instrument maintenance log or bench book, whichever is appropriate.

2. <u>System Corrective Action</u> - A written request form is generated by the QA Officer and is forwarded to the appropriate section supervisor. Either the supervisor or/and analyst assigned by the supervisor will be responsible for the investigation of the problem and the determination of the corrective action required. Investigation may involve evaluation of reagents, acids, extraction solvents, possible sources of contamination, etc.

12.4 Documentation of Problem, Corrective Action and Final Outcome

When the source or sources of the problem have been identified and satisfactorily corrected, the nature of the problem and the measures used to correct the problem are described on the corrective action form. The form is then reviewed, signed and dated by the Section Supervisor and returned to the QA Officer. The QA Officer reviews the returned form to determine if adequate action was taken, signs the form and retains it in the appropriate file. If additional actions are viewed as necessary, the QA Officer returns the form to the section supervisor and the process is repeated.

12.5 Procedures for Review of Corrective Actions

Completion of the corrective action is a sign off of the corrective activity by the laboratory director or an individual assigned to review such actions. Method corrective actions are noted in the instrument logbooks and are initialed and dated by the analyst and/or the supervisor of the section.

Systems corrective action require the final approval of the laboratory director and filed submitted to the Quality Assurance Officer for acceptance and filing with the initial notification of the requirement of a corrective action. The activity of the corrective action is maintained on form **PAS-CAR 186.165 (e) (15)** (see Page 12-3).

Corrective Action Report

Prairie Analytical Systems, Incorporated

Corrective Action Incident Number C____________________

Name ____________________ Title ____________

Signature ____________________ Date ____________

Identification of Problem:

Corrective Action:

Final Outcome:

Name ____________________ Title ____________

Signature ____________________ Date ____________

Review of Corrective Action:

Name ____________________ Title ____________

Signature ____________________ Date ____________

Final Approval:

Note: Corrective action should be performed according to Section 12.0 of the Quality Assurance Plan.

Comments:

13.0 Management Records

13.1 General

Quality assurance reports to management are used to inform senior management personnel of the status of the laboratory operations as related to quality assurance. Reports are prepared by the Quality Assurance Officer and are submitted to the Directors of Prairie Analytical Systems, Inc.

Reports include results of quarterly internal performance and system audits as well as any corrective actions which may have been made as a result of those audits. Additional topics which will appear in reports are new QA procedures or any changes in existing procedures. Significant QA problems and related recommendations will be included when appropriate as well as periodic evaluations of quality control indicators such as accuracy and precision summaries. Any deviations from the requirements of the QAQC Management Plan will also be documented.

These reports to management serve as necessary formal documentation of the Quality Assurance activities of the laboratory. They are also intended as an informative aid to management from which comments and recommendations can be made to the laboratory staff.

1. Inorganics

a. Calibration Verification

1. Initial and final calibration verification is the analysis of the mid-range check standard and blank to verify the analytical system is functional. This is done following initial calibration, immediately prior to any analyses, and following the last analytical sample.

2. Continuing calibration is the analysis of a check standard and blank after every ten (10) analytical samples. It is acceptable to use an initial calibration standard as a continuing calibration check standard.

Acceptance limits for initial and continuing verification are the same.

b. Blanks

1. Trace Metals - Initial blanks are the concentration observed from acidified Type II water. Continuing blanks are measured after every 10 determinations. Preparation blanks are aliquots of pure laboratory water which has been taken through the entire procedural process. Typically, 100 mls laboratory pure water, with acid, will be concentrated to 25 mls and analyzed. One preparation blank is to be analyzed with every twenty (20) samples that are digested.

2. Preparation blanks are specified as to frequency and use after each Inorganic test method. Acceptable limits are detection limits of the test with respect to the sample matrix and size taken for the measurement.

c. Matrix spikes should be at a concentration in the mid-range of the analytical method and be one-half to two times the concentration of the neat sample. Spiked samples, one for each matrix type, should be analyzed. For certain parameters, such as Total Dissolved Solids, negative spiking (a dilution of a sample by a specified amount) may be acceptable. Calculations are identical to positive spikes. The results of all spiked samples are to be reported, regardless of the percent recovery.

d. Lab control samples are spikes into laboratory pure water and are taken through the entire sample preparation. Acceptance is measured by percent recovery. Acceptance limits are stated and are set at 80%-120%. If percent recovery falls outside the limits, the samples associated with the laboratory control sample must be re-analyzed, beginning with sample preparation. All analyses for this parameter will cease until the non-compliance is resolved.

e. Duplicate sample analysis, one sample of each matrix type, or one every ten determinations is to be analyzed in duplicate. Acceptance limits are measures by relative percent difference and set at 20%. If the results are less than (<), relative percent difference cannot be calculated, and "NC" is reported. Since this is not useful information, efforts should be made to avoid particularly clean samples for duplicate samples.

f. Instrument detection limit is based on two times the baseline noise, The IDL's are performed each quarter for each instrument used. Each required detection limit must be met by the instrument that can obtain an IDL at that limit or below. *Detection limits may be specified by contract.*

g. Standard Addition - Samples that do not yield acceptable percent recoveries may be analyzed by the Method of Standard Addition (MSA).

h. Interference Check Sample - When analyzing soil matrices and/or drinking water matrix samples by the ICP, an Interference Check Sample (ICS) must be analyzed and recoveries for that sample reported.

2. Organics

a. GC/MS Tuning and Mass Calibration - Tuning of the Mass Spectrometer is verified daily, or every 12 hours, prior to sample analysis by the obtaining the spectra of either DFTPP or 4-BFB. The actual percent relative abundance is recorded. The date, time of run, operator, and file name area provided as part of the tuning printout.

b. Initial Calibration Check - Retention times and area counts for all compounds in standard runs are documented along with the appropriate file names. The date of the initial calibration is also recorded.

c. Continuing Calibration Verification - Continuing calibration runs are documented. Retention times and percent recoveries for all compounds are recorded along with the date and concentration level. Any calibration of retention time or response is noted. All recoveries outside the acceptable limits are flagged with the appropriate mark.

d. Reagent Blank Summary - Any compounds in the target list detected in a procedural blank above the proposed limits, will be reported on the appropriate procedural summary sheet. The date of analysis, file, name of run, matrix, concentration detected and suspected source of contamination will be recorded.

e. Spike/Spike Duplicate Recovery - All matrix duplicate spike results are recorded. Recovery and duplicate recovery data are recorded and any values outside the acceptance limits are flagged with an asterisk. The date the analysis was performed and the sample description are also documented.

f. Surrogate Percent Recovery - Surrogate recovery for every sample, standard and blank are recorded. The date of analysis, name of file and the laboratory sample number is also recorded. Any value outside the acceptance limits is noted with an asterisk.

13.2 Record Retention[1]

The laboratory documents and maintain records related to all procedures and activities to which a sample is subjected including:

1. identity of personnel involved in sampling, preparation and testing;

2. sample preservation, sample container and compliance to holding times;

3. sample identification code, receipt, log-in, acceptance and rejectance;

4. sample storage and tracking including: shipping receipts, transmittal forms and internal routing, internal laboratory transfer sheets and assignment records;

5. sample preparation including: cleanup and separation procedures, extract or digest identification codes, volumes, weights, instrument printouts, meter readings, calculations and reagents;

6. sample analysis;

7. equipment receipt, use, specification, operating conditions and preventative maintenance;

8. calculations and statistical formulae used by the laboratory including

    a. written procedures for all calculations;

    b. representative calculations that indicate routine calculations;

    c. all raw data and supporting information needed to recreate calculation;

    d. appropriate number of significant digits are carried out throughout all recorded calculations; and

    e. the least precise step is identified in the calculation and the number of significant figures is an accurate reflection of the actual tolerances of the instrument or equipment;

9. procedures to verify that the reported data is free from transcription and calculation errors;

10. data handling;

11. QC measurements, including: reduction, review, confirmation, interpretation, assessment and assessment of method performance;

12. requirements specified in Section 186.185(j) of 35 IAC Part 186.

13. all information necessary to produce unequivocal , accurate records that document the laboratory activities associated with the sample receipt, preparation, analysis and reporting;

14. all information necessary to produce unequivocal link with the unique field identification and the laboratory identification code assigned each sample.

The laboratory retains all of the following records:

1. all original raw data. Wether hard copy or electronic, for calibrations, samples and quality control measures, including analyts' work sheets and data output records such as chromatograms, strip charts and other instrument response readout records;

2. copies of final reports;

3. archived SOPs;

4. all correspondence between the laboratory and the laboratory's clients;

5. all corrective action reports, audits and audit responses;

6. PE sample results and raw data; and

7. data review and cross checking.

The laboratory shall retain all records:

1. Pertaining to drinking water analyses that are associated with the laboratory's accreditation for a minimum of 10 years. Analysis of lead and copper shall be maintained for a minimum of 12 years.

2. Pertaining to environmental analysis that are associated with the laboratory's Accreditation for a minimum of five years unless otherwise designated for a longer period of time in another regulation.

3. Pertaining to all suppliers from whom it obtains support services or suppliers required for tests for a minimum of five years.

14.0 Customer Relations

14.1 General

Quality assurance includes managed response to clients, both public and private, for concerns about results, reporting, requests for report copies, etc. Further, it includes client complaints, written or verbal, regarding personnel and/or general office practices. It also includes the notification of clients of the changes in methodologies that affect sampling procedures, reporting, and other laboratory practices required by USEPA And the IEPA to insure compliance by both the laboratory and the client.

14.2 Procedures for Dealing with Complaints

All requests or complaints filed with the laboratory, either written or verbal, usually involve the reported results of an analysis or the request for copies of a report by someone other then the client for whom the report was prepared. All technical complaints are given to the laboratory director immediately upon receipt by the laboratory.

Following the receipt of the client requests, the laboratory director re-validates all data with the analyst responsible for the analysis of the subject samples to insure that raw data calculations and/or the report is accurate. If the data is in control, the sample log-in procedure is rechecked to insure that the samples received by the lab were logged-in correctly as labeled on the chain-of-custody accompanying the samples in question. Following the re-validation by the laboratory, and within eight hours of the receipt of the complaint, the sample owner is contacted and advised of the the findings of the re-validation of the results. Once determined that the original data generated during the analysis of a sample and the reporting of that data has been properly validated and presented, the client is contacted with the results of the laboratory's findings. Further, the customer is advised a re-verification of "in-field" sampling procedures be in order in a effort to properly match analytical results with the conditions sampled in the field.

14.3 Procedures for Protecting Confidentiality and Proprietary Rights

It is the policy of the laboratory that all analytical data produced by the laboratory and the reports generated as the result of those analysis are confidential. Release of any analytical data or report documents to someone other than the client listed on the chain-of-custody will only be provided following a written authorization by the client or by the mandate of the courts via a record subpoena and the approval of the laboratory director or an officer of the company.

15.0 Bibliography

15.1 Bibliography

1. **Title 35 Illinois Admnistrative Code, Chapter II, Part 186 - Accreditation of Laboratories for Drinking Water, Wastewater and Hazardous waste.** March 1998.

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   40 CFR 136.5(1997),
   40 CFR 136 Appendix A (1997),
   40 CFR 136 Appendix B (1997),
   40 CFR 136 Appendix C (1997),
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   40 CFR 141.24(e) (1997),
   40 CFR 141.27 (1997), and
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8. EPA No. 821-C-97-001, **" Methods and Guidance for Analysis of Water"** Version 1.0, April 1997. U.S. Environmental Protection Agency, Analytical Methods Staff, Engineering and Analysis Division, Washington, DC 20460.

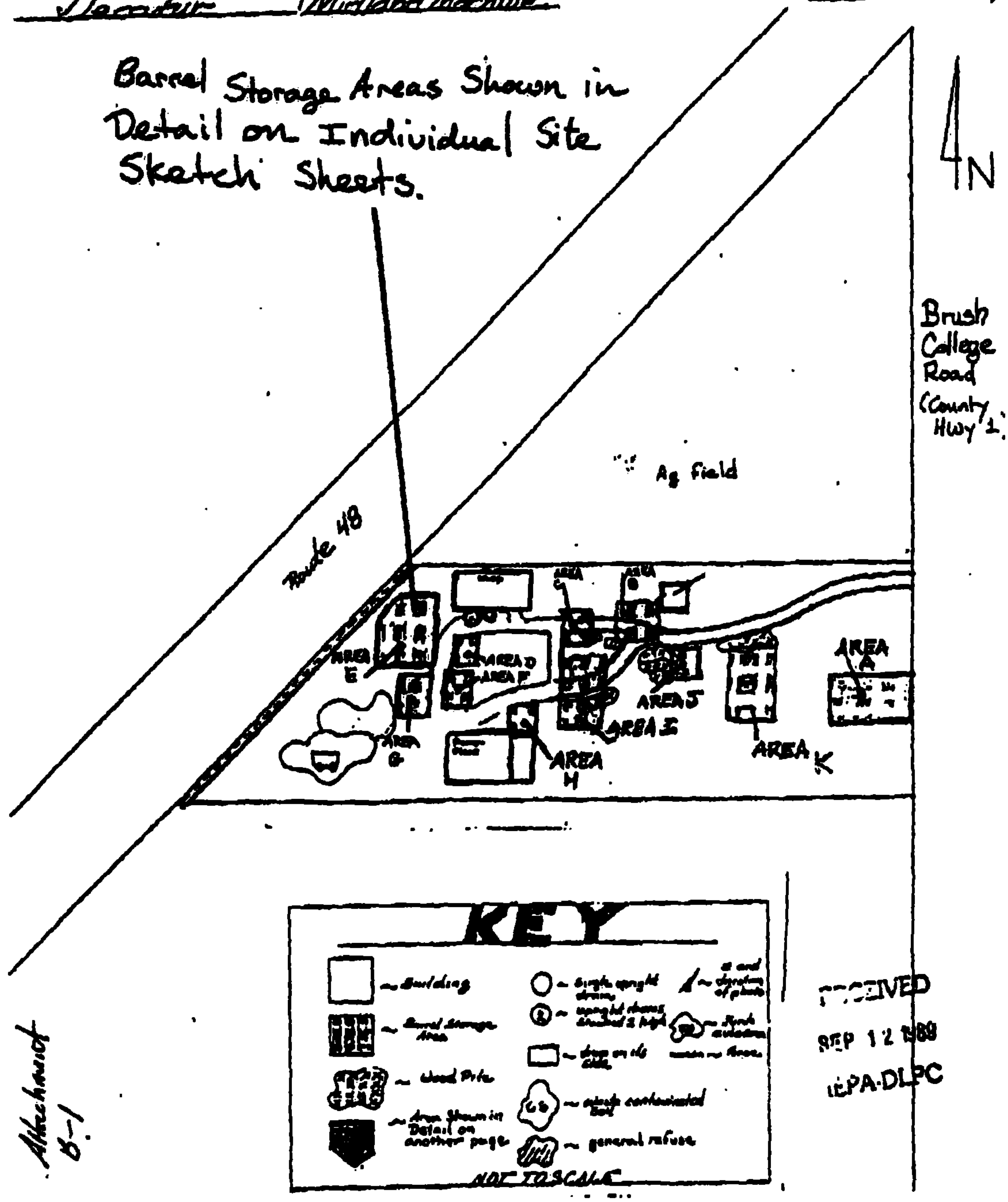
9. **Methods OA-1 & OA-2**, UST Program, Environmental Protection Division, Iowa Department of Natural Resources, Des Moines, IA. Rev. July 1991

ATTACHMENT I

FACILITY SITE SKETCH

Macon · LPC 1150150053 DATE: August 7 1989

Decatur / Midland Machine TIME: 8:00am to 9:00am

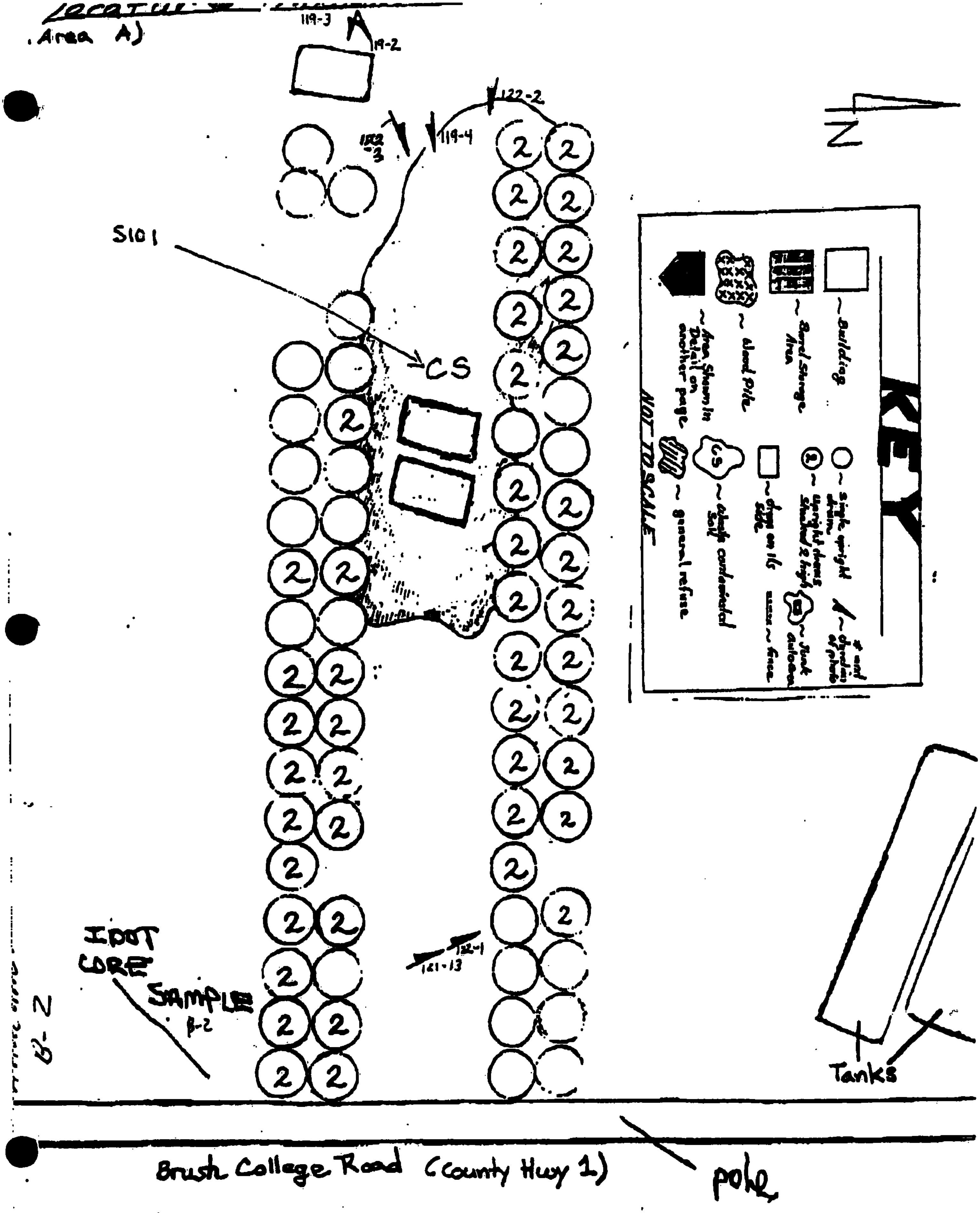


Attachment B-1

RECEIVED
SEP 12 1989
IEPA-DLPC

ATTACHMENT II

AREA "A" SITE SKETCH


Area A)
119-3
119-2
122-2
122-3
119-4
S101
CS
KEY
~ Building
~ Barrel Storage Area
~ Wood Pile
~ Area Shown in Detail on another page
~ single upright drum
~ Upright drums stacked 2 high
~ drums on its side
~ waste contaminated soil
~ general refuse
~ # and direction of photo
~ Junk automobile
~ fence
NOT TO SCALE
122-1
121-13
IDOT CORE SAMPLE
B-2
N
Tanks
Brush College Road (County Hwy 1)
pole